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1 Sedimentary cannabiniol tracks the history of hemp retting

2

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19

20 **ABSTRACT**

21 Hemp (*Cannabis* sp.) has been a fundamental plant for the development of human
22 societies. Its fibers have long been used for textiles and rope making, which requires prior

23 stem retting. This process is essential for extracting fibers from the stem of the plant, but can
24 adversely affect the quality of surface waters. The history of human activities related to
25 hemp (its domestication, spread, and processing) is frequently reconstructed from seeds and
26 pollen detected in archaeological sites or in sedimentary archives, but this method does not
27 always make it possible to ascertain whether retting took place. Hemp is also known to
28 contain phytocannabinoids, a type of chemicals that is specific to the plant. Here we report
29 on the detection of one of these chemicals, cannabinal (CBN), preserved in a sediment
30 record from a lake in the French Massif Central covering the past 1800 yr. The presence of
31 this molecule in the sedimentary record is related to retting. Analysis of the evolution of
32 CBN concentrations shows that hemp retting was a significant activity in the area until ca.
33 A.D. 1850. These findings, supported by pollen analyses and historical data, show that this
34 novel sedimentary tracer can help to better constrain past impacts of human activities on the
35 environment.

36

37 **INTRODUCTION**

38 Hemp is one of the earliest cultivated plants (Russo, 2007). Its high adaptability
39 (Raman, 1998) allowed it to spread worldwide, perhaps through a co-evolution with
40 mankind (McPartland and Guy, 2004). Hemp can therefore be considered as a fundamental
41 plant in human history (Raman, 1998). Indeed the development of all civilizations has relied
42 on its many uses: e.g., as foodstuff (the seeds), medicine and intoxication (the resin), and
43 overwhelmingly for making ropes, textile, and paper (the fibers). The fibers are separated
44 from the stems after the retting process, usually consisting of submerging the stems in water
45 (Wills, 1998). Though retting is required for extracting fibers and is thus used worldwide,

46 this traditional process has been known for centuries to dramatically damage water quality,
47 causing massive fish death and making water undrinkable for cattle and humans
48 (Anonymous, 1772). Tracing this ancient pollution is of major interest, because it can
49 provide clues to past interactions between human societies and environments, the
50 understanding of which is crucial to anticipate the consequences of future global changes
51 (Dearing, 2006). To date, pollen, seeds, or textile fragments are the only indicators currently
52 exploitable in archaeological studies to detect the use of retting (e.g., Schofield and Waller,
53 2005), and thus to assess the extent of the induced pollution.

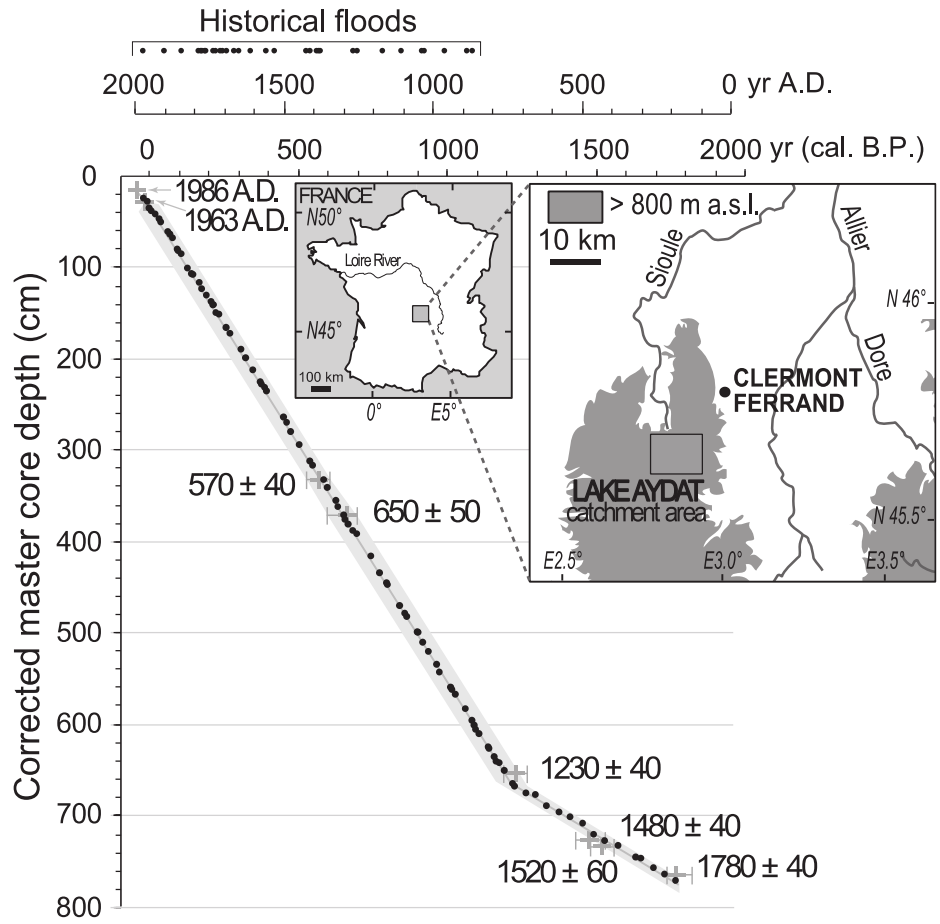
54 Recording a continuous history of hemp retting can be achieved by using a set of
55 archaeological sites that are chronologically continuous and where seeds and pollen are
56 preserved, and implies morphometric analyses of pollen due to the resemblance between
57 *Cannabis* and *Humulus* (hop genus) pollen grains (Mercuri et al., 2002; Whittington and
58 Gordon, 1987). Moreover, pollen and seeds may be absent in archaeological sites (Wills,
59 1998). Monitoring tracers of human activity preserved in a natural archive can help
60 overcome these difficulties. The analysis of the molecular biomarker content of lacustrine
61 sediments (which continuously record environmental changes) and soils can provide
62 information on past environments, but only in a very few cases can these be unequivocally
63 related to human activity (e.g., Bull et al., 2002; Jacob et al., 2005, 2008; Zocatelli et al.,
64 2010; Lavrieux et al., 2011; Le Milbeau et al., 2013). Up to now, no study has revealed the
65 occurrence of any hemp biomarker in natural archives, although the plant contains
66 phytocannabinoids, a group of chemical compounds unique to this plant (Russo, 2007). We
67 here report on the detection of cannabitol in a sediment core drilled in Lake Aydat
68 (Auvergne region, France) that covers the past 1800 yr.

69

70 **METHODS AND STUDY SITE**

71 **Sedimentary Core**

72 Lake Aydat (45°39.809'N, 2°59.106'E) is located in the northern part of the French
73 Massif Central (Fig. 1), a volcanic region located in the center of France. A high-resolution
74 continuous sediment sequence covering the past 6700 yr retrieved under 14.5 m water depth
75 was dated (accelerator mass spectrometry [AMS] radiocarbon dates, ¹³⁷Cs measurements,
76 and detection of historical flood deposits) and extensively described in a previous study
77 (Lavrieux et al., 2013). The present study focuses on the upper part of the core, consisting of
78 dark and faintly laminated sediment interrupted in many places by flood deposits. Samples
79 were selected in the background sediment, i.e., after the flood events were removed. Their
80 position is displayed together with the depth-age model in Figure 1.



81

82 Figure 1. Location of study site in France, depth-age model (Lavrieux et al., 2013), and
 83 position of sediment samples along sedimentary record. Corrected master core depth means
 84 that flood deposits were removed. Accelerator mass spectrometry radiocarbon dates are
 85 reported as calibrated years B.P. (before 1950) and shown as gray crosses and error bars.
 86 Gray crosses without error bars are ^{137}Cs dates. Historical floods are shown above the
 87 graphics. Reconstructed depth-age model is shown with a dark gray line (light gray-margin
 88 of error). Sediment samples are symbolized with black dots. a.s.l.-above sea level.

89

90 **Pollen Analyses**

91 Pollen analyses were performed on 50 samples, spaced 2.5 cm apart, covering the
92 past 1800 yr (intervals of 35 yr). Samples were prepared using standard procedure (Faegri
93 and Iversen, 1989) at the Institut Méditerranéen de Biodiversité et d'Ecologie Marine et
94 Continentale (UMR 7263/CNRS, France). Minimum counts of 500 dry land pollen grains
95 per sample were made. Pollen rates were calculated as a percentage of total land pollen
96 excluding hygrophytes, aquatic plants, and fern spores. Morphometric analyses of pollen
97 grains were carried out according to Mercuri et al. (2002). The *Cannabis-Humulus* pollen
98 curve presented here combines the values of *Cannabis-Humulus* pollen type (pollen diameter
99 25–28 μm) and those of *Cannabis* pollen type (diameter $>28 \mu\text{m}$). The frequencies of
100 *Humulus* pollen type (diameter $<25 \mu\text{m}$) are excluded. The detailed pollen counts are
101 provided in the GSA Data Repository¹.

102 **Lipid Analyses**

103 Sixty (60) lacustrine sediment samples covering the past 1800 yr were dried, crushed
104 in a mortar, and sieved at 2 mm. An internal standard was added to ~1 g of sediment, which
105 was solvent extracted by automatic solvent extraction (Dionex Accelerated Solvent
106 Extractor) using a mixture of CH_2Cl_2 :MeOH (9:1 vol/vol). After removal of the
107 solvent under N_2 , the extract was separated into neutral, acidic, and polar fractions on
108 aminopropylbonded silica. The neutral fraction was further separated into aliphatics,
109 aromatics, ethers and esters, ketones and acetates, and alcohols by flash chromatography on
110 a Pasteur pipette filled with activated silica, using a sequence of solvents of increasing
111 polarity. Alcohol fractions were then trimethylsilylated with N,O-bis (trimethylsilyl)

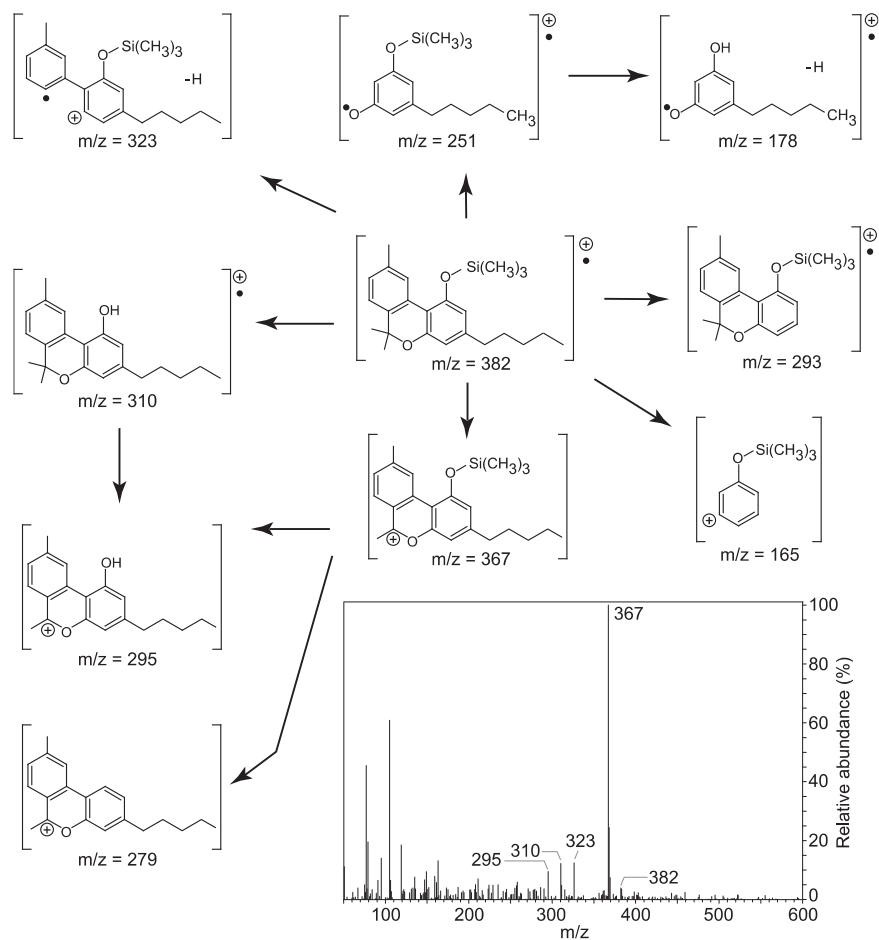
112 trifluoroacetamide and pyridine (2:1 vol/vol; 60 °C, 60 min), and these fractions were
113 injected into a gas chromatography– mass spectrometry (GC-MS) system. The operating
114 conditions are detailed in Lavrieux et al. (2011). Cannabinol (CBN) was identified by
115 comparison with an authentic standard (also trimethylsilylated before injection) and its
116 concentration was estimated by measuring the area of its peak on an m/z 367 + 382 ion-
117 specific chromatogram. After calculating a correction factor between the peak area on the
118 ion-specific chromatogram and the peak area on the total ion current (TIC) chromatogram,
119 the TIC area of the compounds was compared to that of the standard (5 α -cholestane) and to
120 the mass of the sample extracted. The detailed CBN concentrations are provided in the Data
121 Repository.

122

123 **RESULTS**

124 CBN was identified in several alcohol fractions of the free lipids extracted from Lake
125 Aydat sediment samples. Its typical mass spectrum, molecular structure, and fragmentation
126 pattern (as trimethylsilylated derivative) are shown in Figure 2.

127



128

129 Figure 2. Mass spectrum and molecular structure of cannabimol (trimethylsilylated
 130 derivative) from selected sediment sample (A.D. 1322).

131

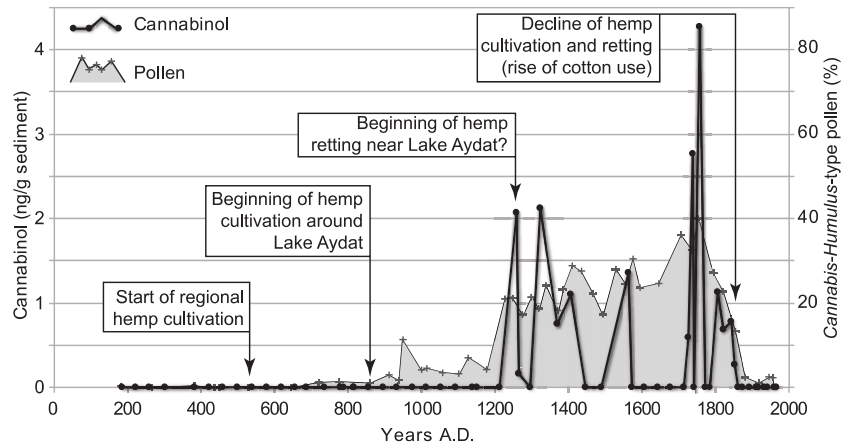
132 This compound is the fully aromatized metabolic by-product of Δ^9 -

133 tetrahydrocannabinol (Δ^9 -THC; ElSohly and Slade, 2005), a psychoactive compound whose

134 concentration in the plant differs depending on the variety of hemp considered (<0.3% in

135 textile hemp versus >1% for intoxicating hemp; Raman, 1998).

136



137

138 Figure 3. Evolution of cannabinol relative abundance (ng/g sediment) and of *Cannabis*-type
 139 and *Cannabis-Humulus*-type pollen (expressed as a percentage of the total of terrestrial
 140 pollen) through time in sediment samples. Main historical phases of hemp uses in the area
 141 are indicated in boxes.

142

143 The concentration of CBN ranged from 0 to 4.27 ng/g sediment in our sediment
 144 samples (Fig. 3). Starting from the base of the studied section, *Cannabis*-type and *Cannabis*-
 145 *Humulus*-type pollen was weakly present from A.D. 470, while CBN was detected for the
 146 first time in sediments dated to ca. A.D. 1260, at concentration levels of ~2 ng/g sediment.
 147 From then on, CBN concentrations and pollen frequency varied strongly, and differently
 148 from each other, all along the record. CBN was not detected in two samples dated from ca.
 149 A.D. 1445–1490 and five samples dated from ca. A.D. 1570–1720, and maximized at A.D.
 150 1757. Pollen frequency followed a more continuous trend, remaining under ~5% until ca.
 151 A.D. 1200, when *Cannabis*-pollen-type values rose to almost 10%, and then increased
 152 abruptly to over 20%. Overall, it remained between ~20% and 30% - with a higher
 153 proportion of *Cannabis* pollen type - until the end of the 18th century, when it maximized at

154 ~40%. From the beginning of the 19th century, both CBN and pollen strongly decreased:
155 CBN was totally absent in sediments younger than ca. A.D. 1860, while pollen was still
156 weakly present (~2%) during the 20th century. CBN was also absent in 35 soil samples
157 analyzed in the lake catchment (Lavrieux, 2011).

158

159 **DISCUSSION**

160 Fiber hemp was extensively cultivated in the Auvergne, France, region during
161 historical times (Peuchet, 1800), and was produced in small plots on the outskirts of every
162 village (Charbonnier, 1980). As in other regions, the main reason for producing hemp fibers
163 was for textile making (de Ballainvilliers, 1846), but all other parts of the plant, except the
164 roots, were used: oil (extracted from seeds) was used as lighting fuel and peeled stems were
165 used for heating (Poitrineau, 1965). Seeds were probably also consumed by local populations
166 and cattle, and leaves were probably used for animal bedding (Brown, 1998). Careful retting
167 was needed to produce a high-quality and reputed hemp fiber such as that used by the French
168 Royal Rope Factory for the marine arsenal, where the longest ropes in Europe (200 m all in
169 one piece) were made during the 17th century (Peuchet, 1800). Hemp was also exported for
170 the paper industry (Peuchet, 1800). Thus, the numerous uses of hemp as well as its
171 widespread culture in the region explain the occurrence of one of its molecular biomarkers in
172 Lake Aydat sediments. Sedimentary CBN could originate either directly from the plant or
173 from hemp remains that were mixed in soils and subsequently eroded to the lake. Because
174 the whole hemp plant was used, most of the material likely to contain CBN was exported.
175 Only roots could constitute a potential contributor of soil CBN, but hemp roots have not
176 been shown to contain more than small amounts of cannabinoids (De Pasquale et al., 1974;

177 Russo, 2007). Because soils are reputed to retain the molecular imprint of their past land
178 uses (Lavrieux et al., 2012), and although soils are the main contributors of terrestrial
179 organic matter to lacustrine sediments through erosion, another source must be invoked to
180 explain the presence of CBN in the lake sediments. The most obvious explanation involves
181 the practice of retting, a process largely used during historical times in Auvergne (Diderot
182 and d'Alembert, 1778; de la Platière, 1784) which consists of submerging the stems in water.
183 This was commonly done in all kinds of aquatic environments such as pits, marshes, ponds,
184 or rivers. The stems were then left in water for a few days to a few weeks in order to
185 facilitate extraction of the hemp fibers.

186 As stated above, pollen analyses conducted on the sediment core show the
187 continuous cultivation of hemp in the region from at least ca. A.D. 470 and in the catchment
188 from at least A.D. 870, and show a strong increase in pollen frequency between A.D. 1180
189 and 1860 (Fig. 3). Conversely, CBN concentration shows a more irregular pattern in this
190 time period and is not detected outside of it. Previous studies in the same catchment (Miras
191 et al., 2004; Lavrieux et al., 2013) revealed continuous human occupation associated with a
192 marked anthropic impact on the environment in the area throughout the time span
193 considered. While the absence of CBN before ca. A.D. 1260 could be explained by limited
194 cultivation (as suggested by the low pollen frequencies) and retting, leading to CBN
195 concentrations that are too low to be detected in the older samples, this hypothesis cannot
196 explain the significant differences observed between pollen and molecular signals for later
197 periods (A.D. 1445– 1490 and 1570–1720). Even though hemp pollen, which is
198 disseminated by wind (e.g., Small and Antle, 2003), could come, in part, from outside the
199 catchment (contrary to CBN, which is necessarily autochthonous), the high frequencies

200 observed during this period leave no doubt concerning the reality of retting near Lake Aydat.
201 No relationship between these discrepancy phases and the different sedimentological
202 parameters expanded by Lavrieux et al. (2013) could be highlighted, underlining that the
203 molecular signal is not determined by the sedimentation rate and/or the dilution in the
204 mineral phase of the sediment. So, in the absence of any other tangible evidence, it can be
205 hypothesized that variations in environmental conditions (for example, intensity of exposure
206 to sunlight and/or of insect predation) known to influence phytocannabinoid concentrations
207 (e.g., Pate, 1994) could have diminished the quantity of CBN in the plant and thus, the
208 quantity archived in the sediment. Further studies are required to test such a hypothesis.

209 Considerable amounts of CBN were still detected throughout the 18th century in our
210 samples, synchronous with maximal values of *Cannabis* pollen rates, despite a royal
211 ordinance dated A.D. 1669 that forbade retting in French rivers in order to preserve water
212 quality, fish stocks, and cattle health (Anonymous, 1772). This observation is in accordance
213 with historical documents, which indicate that this ban was never put into practice and was
214 still being debated 160 yr later (Duvergier, 1830).

215 While the first occurrence of CBN does not correlate with the first occurrence of
216 *Cannabis* sp. pollen in sediments, the abundance of these tracers both strongly decrease in
217 sediments younger than A.D. 1860. On the worldwide scale, this period corresponds to the
218 development of the cheaper cotton industry (e.g., May and Lege, 1999) (followed by
219 synthetic textiles), which hastened the abandonment of textile hemp cultivation.

220

221 **CONCLUSIONS**

222 Consistent with pollen analyses, the presence of CBN in Lake Aydat sediment
223 samples during the period of hemp cultivation in the region strongly suggests that this
224 compound—an unequivocal molecular biomarker of *Cannabis* sp.—can be used in
225 sediments as a sedimentary tracer of anthropogenic activity and pollution for archaeological
226 and paleoecological studies. In addition to pollen studies reflecting the cultivation of hemp,
227 our results indicate that CBN tracks more specifically the subsequent retting process, reputed
228 to significantly alter water quality.

229 Although further work is necessary to better evaluate the stability of CBN in older
230 sediments, this compound can be tracked in natural archives to reconstruct hemp retting
231 history and its induced pollution in a continuous time frame, as opposed to archaeological
232 studies classically performed on archaeological sites that are more constrained in space and
233 time, and can give reliable information about past impacts of human activities on the
234 environment.

235

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243

244 **REFERENCES CITED**

245 Anonymous, 1772, Commentaire sur l'ordonnance des eaux et forêts, du mois d'août 1669:

246 Paris, Debure l'aîné, 516 p.

247 Brown, D.T., 1998, Non-medicinal uses of *Cannabis sativa*, in Brown, D.T., ed., *Cannabis*:

248 The genus *Cannabis*: Amsterdam, Harwood Academic Publishers, p. 115–124.

249 Bull, I.D., Lockheart, M.J., Elhmmali, M.M., Roberts, D.J., and Evershed, R.P., 2002, The

250 origin of faeces by means of biomarker detection: *Environment International*, v. 27,

251 p. 647–654, doi:10.1016/S0160-4120(01)00124-6.

252 Charbonnier, P., 1980, Une autre France: La seigneurie rurale en Basse-Auvergne du XIV^e

253 au XVI^e siècle [Ph.D. thesis]: Clermont-Ferrand, France, University of Clermont-

254 Ferrand, 1294 p.

255 Dearing, J.A., 2006, Climate-human-environment interactions: Resolving our past: *Climate*

256 of the Past, v. 2, p. 187–203, doi:10.5194/cp-2-187-2006.

257 de Ballainvilliers, S.C.S.B., 1846, *Etat de l'Auvergne en 1765*: Clermont-Ferrand, France,

258 Bouillet, 201 p.

259 de la Platière, R., 1784, *Encyclopédie méthodique, Manufacture et arts, Tome second*: Paris,

260 Panckoucke, 315 p.

261 De Pasquale, A., Tumino, G., and Costa De Pasquale, R., 1974, Micromorphology of the

262 epidermic surfaces of female plants of *Cannabis sativa* L.: *Bulletin on Narcotics*, v. 26,

263 p. 27-40.

264 Diderot, D., and d'Alembert, J.L.R., 1778, *Encyclopédie, ou Dictionnaire raisonné des*

265 sciences, des arts et des métiers, Tome septième: Geneva, Switzerland, Pellet, 894 p.

266 Duvergier, J.B., 1830, Collection complète des lois, décrets, ordonnances, réglemens et avis
267 du Conseil-d'Etat (de 1788 à 1824 inclusivement, par ordre chronologique): Paris,
268 Guyot, Scribe et Charles-Béchet, 872 p.

269 ElSohly, M.A., and Slade, D., 2005, Chemical constituents of marijuana: The complex
270 mixture of natural cannabinoids: *Life Sciences*, v. 78, p. 539–548,
271 doi:10.1016/j.lfs.2005.09.011.

272 Faegri, K., and Iversen, J., 1989, Textbook of pollen analysis, 4th edition: Caldwell, New
273 Jersey, The Blackburn Press, 328 p.

274 Jacob, J., Disnar, J.R., Boussafir, M., Albuquerque, A.L.S., Sifeddine, A., and Turcq, B.,
275 2005, Pentacyclic triterpene methyl ethers in recent lacustrine sediments (Lagoa do
276 Caçó, Brazil): *Organic Geochemistry*, v. 36, p. 449–461,
277 doi:10.1016/j.orggeochem.2004.09.005.

278 Jacob, J., Disnar, J.R., Arnaud, F., Chapron, E., Debret, M., Lallier-Vergès, E., Desmet, M.,
279 and Revel-Rolland, M., 2008, Millet cultivation history in the French Alps as evidenced
280 by a sedimentary molecule: *Journal of Archaeological Science*, v. 35, p. 814-820,
281 doi:10.1016/j.jas.2007.06.006.

282 Lavrieux, M., 2011, Biomarqueurs moléculaires d'occupation des sols, du sol au sédiment:
283 Exemple du bassin-versant et du lac d'Aydat (Puy-de-Dôme) [Ph.D. thesis]: Tours,
284 France, Université François-Rabelais de Tours, 246 p.

285 Lavrieux, M., Jacob, J., Le Milbeau, C., Zocatelli, R., Masuda, K., Bréheret, J.G., and
286 Disnar, J.R., 2011, Occurrence of triterpenyl acetates in soil and their potential as
287 chemotaxonomical markers of Asteraceae: *Organic Geochemistry*, v. 42, p. 1315–1323,
288 doi:10.1016/j.orggeochem.2011.09.005.

289 Lavrieux, M., Bréheret, J.G., Disnar, J.R., Jacob, J., Le Milbeau, C., and Zocatelli, R., 2012,
290 Preservation of an ancient grassland biomarker signature in a forest soil from the French
291 Massif Central: *Organic Geochemistry*, v. 51, p. 1–10,
292 doi:10.1016/j.orggeochem.2012.07.003.

293 Lavrieux, M., Disnar, J.R., Chapron, E., Bréheret, J.G., Jacob, J., Miras, Y., Reyss, J.L.,
294 Andrieu-Ponel, V., Arnaud, F., 2013, 6,700-year sedimentary record of climatic and
295 anthropogenic signals in Lake Aydat (French Massif Central): *The Holocene* v. 23,
296 1317-1328. doi:10.1177/0959683613484616

297 Le Milbeau, C., Lavrieux, M., Jacob, J., Bréheret, J.G., Zocatelli, R., and Disnar, J.R., 2013,
298 Methoxy-serratenes in a soil under conifers and their potential use as biomarkers of
299 Pinaceae: *Organic Geochemistry*, v. 55, p. 45–54,
300 doi:10.1016/j.orggeochem.2012.11.008.

301 May, O.L., and Lege, K.E., 1999, Development of the world cotton industry, in Smith, C.
302 W., and Cothren, J.T., eds., *Cotton: Origin, History, Technology and Production*: New
303 York, John Wiley & Sons, p. 65–98.

304 McPartland, J.M., and Guy, G.W., 2004, The evolution of cannabis and coevolution with the
305 cannabinoid receptor—A hypothesis, in Guy, G.W., et al., eds., *The Medicinal Uses of*
306 *Cannabis and Cannabinoids*: London, Pharmaceutical Press, p. 71–102.

307 Mercuri, A.M., Accorsi, C.A., and Mazzanti, M.B., 2002, The long history of *Cannabis* and
308 its cultivation by the Romans in central Italy, shown by pollen records from Lago
309 Albano and Lago di Nemi: *Vegetation History and Archaeobotany*, v. 11, p. 263–276,
310 doi:10.1007/s003340200039.

311 Miras, Y., Laggoun-Défarage, F., Guenet, P., and Richard, H., 2004, Multi-disciplinary
312 approach to changes in agro-pastoral activities since the Sub-Boreal in the surroundings
313 of the “narse d’Espinasse” (Puy de Dôme, French Massif Central): Vegetation History
314 and Archaeobotany, v. 13, p. 91–103, doi:10.1007/s00334-004-0033-z.

315 Pate, D.W., 1994, Chemical ecology of *Cannabis*: Journal of the International Hemp
316 Association, v. 2, p. 32–37.

317 Peuchet, J., 1800, Dictionnaire universel de la géographie commerçante, Tome quatrième:
318 Paris, Blanchon, 797 p.

319 Poitrineau, A., 1965, La vie rurale en Basse-Auvergne au XVIII^e siècle (1726–1789) [Ph.D.
320 thesis]: Paris, University of Paris, 929 p.

321 Raman, A., 1998, The *Cannabis* plant: Botany, cultivation and processing, in Brown, D.T.,
322 ed., *Cannabis: The genus Cannabis*: New York, Harwood Academic Publishers, p. 29–
323 54.

324 Russo, E.B., 2007, History of Cannabis and its preparations in saga, science, and sobriquet:
325 Chemistry & Biodiversity, v. 4, p. 1614–1648, doi:10.1002/cbdv.200790144.

326 Schofield, J.E., and Waller, M.P., 2005, A pollen analytical record for hemp retting from
327 Dungeness Foreland, UK: Journal of Archaeological Science, v. 32, p. 715–726,
328 doi:10.1016/j.jas.2004.12.004.

329 Small, E., and Antle, T., 2003, A preliminary study of pollen dispersal in *Cannabis sativa* in
330 relation to wind direction: Journal of Industrial Hemp, v. 8, p. 37–50,
331 doi:10.1300/J237v08n02_03.

332 Whittington, G., and Gordon, A.D., 1987, The differentiation of the pollen of *Cannabis*
333 *sativa* L. from that of *Humulus lupulus* L.: Pollen et Spores, v. 29, p. 111-120.

334 Wills, S., 1998, *Cannabis* use and abuse by man: An historical perspective, in Brown, D.T.,
335 ed., *Cannabis: The genus Cannabis*: New York, Harwood Academic Publishers, p. 1-27.
336 Zocatelli, R., Jacob, J., Turcq, B., Boussafir, M., Sifeddine, A., and Bernardes, M.C., 2010,
337 Biomarker evidence for recent turf cultivation in Northeast Brazil (Lagoa do Boqueirão,
338 RN State): *Organic Geochemistry*, v. 41, p. 427-430,
339 doi:10.1016/j.orggeochem.2009.12.008.

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341 ¹GSA Data Repository item 2013209, Table DR1 (*Cannabis*-type and *Cannabis-Humulus*-
342 type pollen counts), is available online at www.geosociety.org/pubs/ft2013.htm, or on
343 request from editing@geosociety.org or Documents Secretary, GSA, P.O. Box 9140,
344 Boulder, CO 80301, USA.