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VARIABILITY OF RADIOCARBON RESERVOIR AGE EFFECTS IN LAKES AND RIVERS IN ANATOLIA AND LESSER CAUCASUS

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ABSTRACT. Multiproxy sedimentary sequence analysis constitutes the basis for reconstructions of past paleoenvironments and climate evolution. These sequences are, for the most part, obtained by coring in lakes, maars or crater lakes whose waters can record volcanic activity or karstic contributions, especially in Eastern Anatolia and the Lesser Caucasus. The reservoir age effect in these geological contexts leads to an apparent aging of the radiocarbon ages which also affects the plants and animals developing in or near these waters and consequently the population consuming them. We present here some results obtained from modern samples taken from Mediterranean, central and eastern Anatolian lakes, from the Van and Sevan lakes and along the Kura River and its tributaries from the Lesser Caucasus. The effect of volcanic CO_2 outgassing in the vicinity of maar crater lakes is also discussed.

KEYWORDS: Anatolia, Caucasus, karstic area, reservoir age, volcanic area.

INTRODUCTION

For about forty years, the estimation of reservoir ages to correct the aging of radiocarbon dates has been a major concern of archaeologists and geologists. This reservoir effect results, during the synthesis of the dated organic sample, from the use of carbon whose ^{14}C activity is different from that of contemporary atmospheric CO_2. It is defined as:

$$R(^{14}C\ reservoir\ ages) = {}^{14}Cage\ of\ sample - {}^{14}Cage\ of\ contemporaneous\ atmospheric\ CO_2$$

and is expressed in ^{14}C years (Mangerud 1972).

This reservoir effect in non-marine water-bodies is known to have multiple origins: (1) the hard water effect which results mainly from the dissolution of fossil carbonate rocks; (2) degassing of volcanic CO_2 in the atmosphere or in lakes; or even (3) confinement (cessation of exchanges with the atmosphere). These inputs lead to an aging of lacustrine or underground waters, inducing a reservoir age effect and biasing the radiocarbon dates of the palaeoclimatological record from such environments (Deevey et al. 1954; Deevey and Stuiver 1964). As the original source of carbon for all the aquatic trophic chain, the depleted ^{14}C signature of Dissolved Inorganic Carbon (DIC) is reflected in all the halieutic resources of these lakes. This reservoir effect then propagates in terrestrial animals and humans consuming these food resources, and is reflected in the skeleton of the lake population (Oana and Deevey 1960; Philippsen and Heinemeier 2013, among others). In Asia Minor and the Caucasus, these three aging processes are frequent and are sometimes cumulative.

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While the inland seas (Black and Caspian seas) (see among others Jones and Gagnon 1994; Kuzmin et al. 2007; Fontugne et al. 2009) and the northern Greater Caucasus in Russia (Shishlina et al. 2007; Higham et al. 2010) have already been partially studied, practically no age reservoir data have been published for the Lesser Caucasus and Anatolia. Here, we present age reservoir results obtained between 1999 and 2014. The spatial resolution of the results on the regional to local scales is somewhat incomplete, mainly because some of these regions (e.g. Armenia, Georgia, Azerbaijan, Eastern Taurus, etc.) have been or are still, depending on the period, inaccessible due to recurrent armed conflicts.

MATERIAL AND METHODS

The samples were collected between 1999 and 2014 during different scientific projects in Turkey and the Caucasus. The location, the nature of the samples and their date of collection are reported in Table 1 and Figure 1.

Chemical Treatment and Conversion to CO_2

Water Samples

After collection, surface water samples were immediately poisoned with mercury chloride and stored hermetically in 250 mL glass bottles until laboratory analysis was performed according to the methods described by Duplessy (1972). In the laboratory, water samples between 50 and 100 mL were introduced in a vacuum line, acidified with phosphoric acid, and bubbled with helium gas in order to optimize the extraction of the total dissolved carbon dioxide (Bard *et al.* 1988). The evolved CO_2 was purified, trapped and quantified. An aliquot of CO_2 gas was also sampled to measure the stable carbon isotopic composition ($\delta^{13}C$) with a Fisons-OPTIMA mass spectrometer. The precision was better than 0.05‰.

Organic Samples

Fish samples were bought from fishermen fishing by the river. Fish bone and flesh were dried and stored at –18°C. Fish were collected as part of a paleodietary study using stable isotopy. Collagen was extracted according to the procedures in use in this community (Dufour et al. 1999), i.e. including a lipid elimination step (solvent extraction (Bligh and Dyer 1959); no chemical treatment was carried out on fish flesh. Reed and acacia leaves were dried as soon as possible after collection and were processed according to the standard acid-alkali-acid (AAA) treatment, i.e. 1M HCl, 0.1M then 1M NaOH, and 0.1M and 1M HCl. All treatments were performed at room temperature either in an ultrasonic bath or under agitation. Rinsing with ultrapure water followed each step.

About 1 mg of clean organic sample was then sealed in a quartz tube under vacuum with an excess of copper oxide and silver wire. The tubes were placed in an oven at 840°C for 5 hours to transform the organic matter into CO_2.

CO_2 Reduction and Measurements

Evolved CO_2 was either sent to the Gif-sur-Yvette Accelerator Mass Spectrometry (AMS) ^{14}C laboratory (samples numbered GifA, Arnold et al. 1989) that operated a Tandetron at that time or to the French national AMS facility in Saclay (LMC14, samples numbered SacA-, Cottereau et al. 2007) that operates a NEC AMS. CO_2 was thus graphitized according to protocols slightly modified from Arnold et al. (1989) and measured on AMS. See Dumoulin

Table 1 List of samples used to constrain reservoir age variability. The first three columns give the sample description, collection year and location. The last four columns concern laboratory handling: i – the fraction that was extracted from the sample for ^{14}C analysis, ii – lab identification for measurement and chemical treatment (if only one number, identification is for both chemistry and measurement); please note that the numbers of samples processed in 1999 by the Tandetron team are lost and cannot be recovered because the database crashed, iii – the ^{14}C age, and iv – the R that is calculated deriving from the ^{14}C atmospheric data of the collection year as provided by Hua et al. (2022) for the NH2 zone.

Sample identification							^{14}C data			
Sample N°	Location	Latitude	Longitude	Sample origin	Collection year	Type of sample	Analyzed fraction	Lab identification	Conventional age ± 1 sigma (BP)	R ** (^{14}C yr)
Antalya region										
1	Antalya (DSI PETROL)	36°55'17"N	30°40'35"E	Water table: (well -80m) (water pumping station)	2006	water	DIC*	SacA 5790	8250 ± 40	8730 ± 45
2	Öküzini marshes	37°06'02"N	30°35'04"E	Kırkgözler (spring-fed lake north of ÖKüzini)	2006	water	DIC	SacA 5791	15810 ± 60	16290 ± 65
3	Öküzini marshes	37°04'56"N	30°34'50"E	Karstic source springing in the Öküzini marsh	2006	water	DIC	SacA 5792	17150 ± 80	17630 ± 80
4	Öküzini marshes	37°06'36"N	30°34'50"E	Pınarbaşı (water table in marsh at Plain center)	2006	water	DIC	SacA 5793	13360 ± 50	13840 ± 55
Central Anatolia region										
5	Acıgölü	37°42'44"N	33°40'21"E	Crater Lake (saline)	1999	water	DIC	GifA (1999)	12020 ± 120	12770 ± 120
6	Meke gölü	37°41'24.3"N	33°38'24"E	Crater lake (saline)	1999	water	DIC	GifA (1999)	2150 ± 70	2895 ± 75
7	Karapınar	~38°31'31"N	~34° 32'53.7"E	Water table at Mr Aydınbelge's home (karstic)	1999	water	DIC	GifA (1999)	13770 ± 130	14520 ± 130
8a	Karacaören maar, near Eski Acıgol (1330 m)	38°32'18"N	34°35'28"E	Karacaören (Hot springs) (−80m) near crater lake	1999	water	DIC	GifA (1999)	≥43500	≥43500
8b	Eski Acıgol (1270m)	38°33'01"N	34°32'41"E	see Roberts et al. The Holocene (2001)	1995	modern soil	bulk OM	GrN-23467	1360 ± 30	2265 ± 35
8c	Eski Acıgol (1270m)			see Roberts et al. The Holocene (2001)		Sediment cores	bulk OM	Ra/Th and U/Th ages		3100 ± 35
9	Nar Gölü (1363m)	38°20'16.0"N	34°27'20.8"E	Few meters from the shore of the crater lake	2002	reed	bulk OM	GifA17090/ Gif-11796	1525 ± 30	2150 ± 35

(Continued)

Table 1 (*Continued*)

Sample identification							^{14}C data			
Sample N°	Location	Latitude	Longitude	Sample origin	Collection year	Type of sample	Analyzed fraction	Lab identification	Conventional age ± 1 sigma (BP)	R ** (^{14}C yr)
10	Nar Gölü (1363m)	38°20'12.5"N	34°27'12.1"E	100 meters from the shore of the crater lake	2002	acacia leaves	bulk OM	SacA44568/ Gif-11797	pMC 102 ± 0.3	400 ± 35
11	Nar Gölü (1363m)	38°20'17.5"N	34°27'23.0"E	Crater lake	1999	water	DIC	GifA (1999)	26100 ± 390	26850 ± 390
12	Obruks Plateau	37°35'26.4"N	33°35'50.1"E	Source (Water table in karst)	1999	water	DIC	GifA (1999)	4420 ± 80	5170 ± 80
13	Çora maar (near Erciyes dağ)	38°39'36"N	35°17'59"E	Core Interstitial surface Water in maar (volc)	2006	water	DIC	SacA 5794	1420 ± 30	1900 ± 35
Lake Van										
14	South of the lake (Taurus)	38°26N	42°41E	Collected in the karstic rejection (white plume)	2009	water	DIC	SacA 15095/ Gif-12407	4200 ± 35	4600 ± 40
15	South of the lake (Taurus)			Collected outside the karstic rejection	2009	water	DIC	SacA 15096/ Gif-12408	1935 ± 35	2335 ± 40
16	South of the lake (Taurus)			Collected outside the karstic rejection	2009	water	DIC	SacA 15097/ Gif-12409	1845 ± 30	2245 ± 35
	Center of the lake	38°36'52"N	42°53'04"E	Makaroğlu et al. Quat Int. (2016)		sediment	bulk OM			2500-2800
Armenian Lakes										
17	Lac Yerevan (908m)	40°09'38"N	44°28'43"E	Artificial lake near Erevan city	2014	farm trout bone	collagen	SacA41910/ Gif-13147	20 ± 30	230 ± 35
18	Lac Sevan (Alt 1895m)	~40°34'N	~45°00'E	Lake	2014	wild trout bone	collagen	SacA41912/ Gif-13149	730 ± 30	940 ± 35
19	Lac Sevan (Alt 1895m)	~40°34'N	~45°00'E	Lake	2014	carp bone	collagen	SacA41913/ Gif-13150	665 ± 30	875 ± 35
20	Lac Sevan (Alt 1895m)	40°34'01"N	45°00'28"E	Lake	2014	water	DIC	SacA36821/ GifA-14010/Gif-13072	950 ± 30	1160 ± 35
Kura Valley (Georgia)										
21	Tbilissi - Kura river	41°43'35"N	44°46'53"E	Kura River Near Tbilissi	2014	Laebobarbus (*Varicorhinus Sapoeta*) bone	collagen	SacA41911/ Gif-13148	pMC 102 ± 0,28	0 ± 35

Table 1 (*Continued*)

Sample identification							^{14}C data			
Sample N°	Location	Latitude	Longitude	Sample origin	Collection year	Type of sample	Analyzed fraction	Lab identification	Conventional age ± 1 sigma (BP)	R ** (^{14}C yr)
Kura valley (Azerbaidjan)										
22	Confluence with Agstafa river	41°14'29.2"N	45°26'12.5"E	Geologic bedrock (limestone)	2013	fish flesh	bulk OM	SacA 38572/ Gif-13109	1065 ± 30	1305 ± 35
23	Confluence with Agstafa river	–	–	Geologic bedrock (limestone)	2013	fish flesh	bulk OM	SacA 38573/ Gif-13110	900 ± 30	1140 ± 35
24	Bridge near confluence Tovuz river	41°03'51"N	45°47'41" E	Geologic bedrock (limestone)	2014	fish flesh	bulk OM	SacA40568/ Gif-13127	910 ± 30	1120 ± 35
25	Upper stream of Zeyem river	40°38'19" N	45°39'37" E	Geologic bedrock (volcanic)	2014	fish flesh	bulk OM	SacA40569/ Gif-13128	150 ± 30	360 ± 35
Mil Plain										
26	(near Agiabedi)	39°53'35.7"N	47°25'24.9"E	Irrigation channel from Aras River Geologic bedrock (Caspian Sea and Araxe River sediments)	2013	fish flesh	bulk OM	SacA 38574/ Gif-13111	205 ± 30	445 ± 35

*DIC dissolved inorganic carbon,

**R calculated using Hua et al. (2022) to know the atmospheric ^{14}C content of the collection year

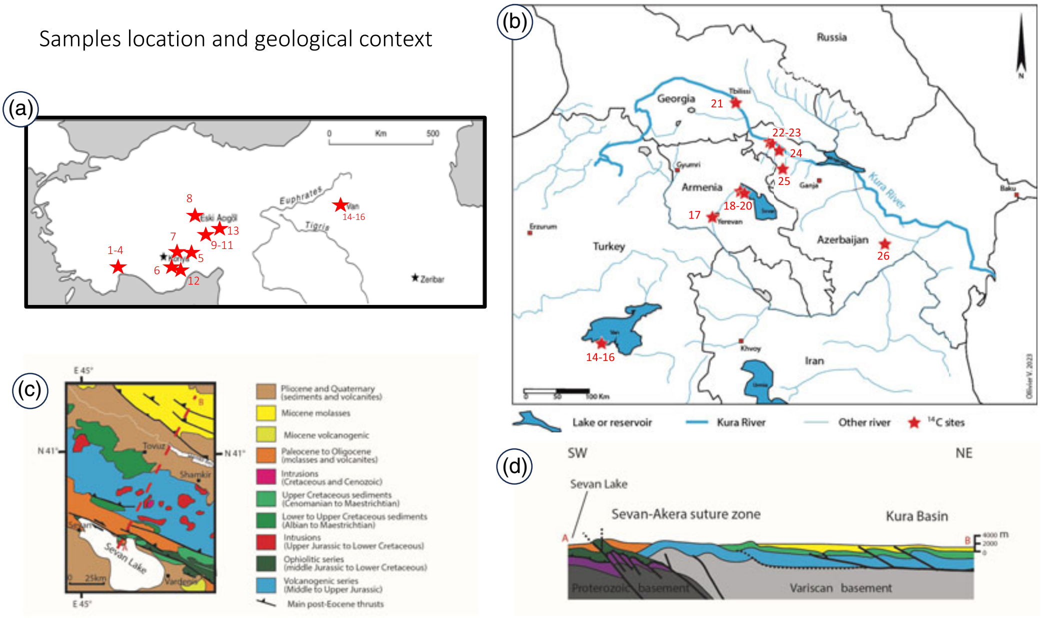

Figure 1 Location and geological context of the samples. 1a – Map of the Turkish region that shows the location of samples 1 to 16. 1b – Map of the Kura River and tributaries, lesser Caucasus and Eastern Anatolia (adapted from Ollivier et al. 2016, 2018). The red stars point to the location of the analyzed samples listed in Table 1. 1c – Geological map and 1d – profile of the Lesser Caucasus in the studied area (adapted from Ollivier et al. 2016 and Sosson et al. 2010). The dashed red line in 1c shows the AB section of the profile shown in 1d.

et al. (2017) and Moreau et al. (2020) for updated details at LMC14; Hatté et al. (2023), Tisnérat-Laborde et al. (2015) and Thil et al. (submitted) for updated details at LSCE.

Results are expressed as conventional ^{14}C age, rounded up as recommended by Stuiver and Polach (1977). The atmospheric ^{14}C age values were calculated using "Atmospheric ^{14}C age = -8033*ln(F^{14}C)". All samples belong to NH zone 2 defined in Hua et al. (2022 and suppl. mat.). The average value of F^{14}C of the year of collection was used for calculation. Reservoir age was calculated thanks to the equation previously reported. All results are reported in Table 1.

RESULTS AND DISCUSSION

First of all, it is worth pointing out that not all reservoir ages have the same meaning. Thus, those obtained on organic samples give a more integrated average value while the samples of dissolved bicarbonate (DIC) are more or less punctual, reflecting a degree of re-equilibration of the mineral carbon dissolved in these waters with the atmosphere. Furthermore, the reservoir age may be underestimated due to the variable input of bomb ^{14}C stored in soils and peats in lakes and rivers (Marchenko et al. 2021). Alternatively, it may be overestimated due to artificially high atmospheric values that are not yet in equilibrium with the old carbon reservoir (although the most recent samples (e.g. after 2000 AD) will be minimally affected).

The Antalya Region (Turkey)

The results are reported in Table 1. The Öküzini marshes extend at the edge of the Antalya Plain travertines, at the foot of the Western Taurus thick limestone massif (Kuzucuoğlu *et al.* 2001). Samples 2 to 4 taken near the springs feeding the marsh show reservoir ages varying between 13,800 and 17,600 ^{14}C years. Further south, water from a well (sample 1) coming from the aquifer delivers a reservoir age of 8730 ± 45 ^{14}C years which records the partial re-equilibration of these waters with the atmosphere.

The Central Anatolia Region (Turkey)

In a limestone and volcanic context, the waters of the aquifer also have variable reservoir ages: from 5170 ± 80 ^{14}C years for sample 12, from the Obruks Plateau (doline lake in the limestone context north of Karapinar, Kuzucuoğlu 2019), to 14,520 ± 130 ^{14}C years for a deep aquifer well at Karapinar (sample 7).

On the Anatolian Plateau, many volcanic edifices are associated with crater lakes, three of which were sampled (5, 6, 11). The surface water reservoir ages are extremely variable: 2895 ± 75 years for Meke Gölü (sample 6, basaltic maar, south of Karapinar), 12,770 ± 120 ^{14}C years for Acigölü (sample 5, basaltic maar, east of Karapinar, most probably Holocene) and 26,850 ± 390 ^{14}C years for Nar Gölü (sample 11, basaltic maar, north of the Göllüdağ massif). The high ages in Karapinar and Göllüdağ regions in fact record the CO_2 degassing of a volcanic edifice in the lake waters. Such outgassing is usually recorded by plants growing nearby (e.g. Pasquier-Cardin et al. (1999)). In the Nar Gölü crater which extends to the bottom of a basin bordered by the walls of the crater, about a hundred meters high, this degassing also marks the vegetation of the banks with reservoir ages decreasing rapidly from 2150 ± 35 ^{14}C years for the reeds a few meters from the shore of the lake (sample 9) to 400 ± 35 ^{14}C years for the leaves of the acacia 100 m further away (sample 10).

Further east, near the Erciyes volcano, the interstitial waters of the Ḉora maar (basaltic) give an age of 1900 ± 35 ^{14}C years (Sample 13) used by Gauthier et al. (2014) for pollen sequence chronology.

To the east of the Eski Acigöl crater (rhyolitic maar, northern Cappadocia, Kuzucuoğlu et al. 1998), the Karacaören hot springs (sample 8a) have waters devoid of ^{14}C. By their degassing and their contribution to the swamp in the center of the crater, the hot springs are at the origin of reservoir effects. Roberts et al. (2001) mentioned a modern soil age of 1630 BP, meaning a reservoir age of 2265 ± 35 ^{14}C years (sample 8b). In the same study, Roberts et al. calculated, from Ra/Th and U/Th dating along sediment cores taken from the swamp, a reservoir age of 3100 ± 35 ^{14}C years that has remained fairly constant for the last 16 millennia (sample 8c). The reservoir age difference between the 3100 ^{14}C years offset for the sediment core and that of 2265 ^{14}C years for soil may be due to a variable input of bomb ^{14}C, stored in soils and peats, into lakes and rivers in agreement with the observations by Marchenko et al. (2021) in Western Siberia.

The Eastern Anatolia Region (Turkey)

Lake Van is bordered to the north by the volcanic complexes of Nemrut Dag and Süphan Dag and to the south by the limestone massif of the Eastern Taurus mountains. Lake Van is a large soda lake, with a hyper-alkalinity of the water explained by discharges of strongly mineral deposits related to volcanic and hydrothermal activity, to which are added the carbonated contributions by karst inputs from the Taurus Mountains and rivers (Kuzucuoglu *et al.* 2010). Three samples were taken from the south of the lake: sample 14, which comes from a karst water discharge materialized in the lake by a whitish plume resulting from carbonate precipitation, has a reservoir age of 4600 ± 40 ^{14}C years; samples 15 and 16 taken outside this karstic discharge give similar results, 2335 ± 40 and 2245 ± 35 ^{14}C years, respectively. These intermittent karstic discharges linked to episodes of rain on the Taurus mountains are likely to increase the hard water effect but their influence remains limited to small areas to the south of the lake.

Previous work carried out on sedimentary cores and dated from varve counts made it possible to calculate reservoir ages. Lemcke (1996) and Kempe et al. (2002) working on cores retrieved in the center of the deepest depression of the lake (western part), estimated reservoir ages ranging from 2600 years in the surface sediment to about 4700 years in the Late Glacial. From cores extracted in the north of the same depression and at a lower depth, Makaroglu et al. (2016) calculated an average reservoir age of between 2500 and 2800 years for sediments collected from the central-southern part of the lake (lower than the previous one). Our measurements are in good agreement with these studies and thus reinforce the confidence in the estimates obtained by the varve counts. Unfortunately, the eastern part of the lake, where the rivers allow the development of greater biological activity at their mouth, has not yet been documented.

The Armenian Lakes

In Armenia, Lake Sevan, despite its resemblance to the volcanic environment of Lake Van, is not a sodic lake, thus allowing the development of a flourishing aquatic life. The water sample (20) has an age of 1160 ± 35 ^{14}C years which is almost entirely recorded in the flesh of a carp (Sample 19: 875 ± 35 ^{14}C years) and a trout (Sample 18, 940 ± 35 ^{14}C years) living in the western part of the lake. On the other hand, near Yerevan city, the artificial lake devoted to trout farming does not seem to be subject to a notable volcanic influence, as indicated by the age of sample 17 (trout flesh, 230 ± 35 ^{14}C years).

The Kura Valley (Georgia and Azerbaijan) and the Plain of Mil (Azerbaijan)

This variability of reservoir ages is also noted in Georgia and Azerbaijan for the Kura River and its tributaries (Samples 21 to 26). In a volcanic context, fish from the Kura in Tbilisi and that from Zeyem Caye indicate fairly low reservoir ages of 0 ± 35 ^{14}C years and 360 ± 35 ^{14}C years, respectively. Conversely, fish sampled in the Kura River upstream from the mouth of the Zeyem caye show significantly higher ages: 1305 ± 35, 1140 ± 35 and 1120 ± 35 ^{14}C years. These ages result from the exchanges of the Kura River with the water table in the Cretaceous limestones which constitute the bed of the Kura in this region (Figure 1b–d). Such an effect has been described in detail by Coularis *et al.* (2016) for the Loire and its tributaries. In the plain of Mil, on a substrate consisting of sediment from the Caspian Sea and the Araxes River, fish from an irrigation canal give an intermediate age of 445 ± 35 ^{14}C years, in good agreement with previous observations.

CONCLUSION

The aging of the radiocarbon ages of the waters of the Anatolian lakes or the Lesser Caucasus is a general phenomenon resulting from volcanic activity (Central and Eastern Anatolia) and/

or from karstic discharges from the Taurus mountains (Öküzini in Mediterranean Anatolia, Central Anatolian volcanic province, Lake Van region) and from the Tertiary limestone basement in Central Anatolia. In the crater lake where it has been measured (Nar Gölü), the vegetation in the immediate surroundings records the degassing of the waters. These effects of degassing are observed in all the sites studied but often concern only restricted areas, located a few tens or hundreds of meters from the emitting source. The low geographical impact was also noted for the karstic discharges south of Lake Van. Such reservoir ages of lake water are almost all recorded by the aquatic fauna as evidenced by the results of Lake Van and Lake Sevan. For the rivers of the Caucasus, the reservoir ages are dependent on the geological substrate: ages on the limestone formations are high compared to those on the granite or volcanic formations, confirming the observations of Coularis *et al.* (2016).

This study shows, through the multiplicity of situations, a great variability in reservoir ages resulting, among other things, from the distance to the source of dead carbon and the level of re-equilibration of the latter with atmospheric CO_2.

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