Reconstruction of the funeral ceremony held before the burial of the king. Tumulus MM is shown under construction in the background.
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APPENDIX 5
CHEMICAL IDENTIFICATION OF THE BEVERAGE
AND FOOD REMAINS IN TUMULUS MM

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Between 1997 and 1999, ancient organic remains contained in 29 of the bronze and pottery vessels from Tumulus MM were analyzed. Most of the chemical studies were carried out in the Molecular Archaeology Laboratory of the Museum Applied Science Center for Archaeology (MASCA) at the University of Pennsylvania Museum, with generous support from The Kaplan Fund of New York and in collaboration with Dr. Donald L. Glusker and Lawrence J. Exner. Selected samples, using a range of scientific techniques not available in MASCA, were also analyzed by the following investigators, with funding from the National Science Foundation and their home institutions and companies: Drs. Robert A. Moreau and Alberto Nuñez (Eastern Regional Research Center, U.S. Dept. of Agriculture, Wyndmoor, PA), Drs. Curt W. Beck and Edith C. Stout (Amber Research Laboratory, Vassar College, Poughkeepsie, NY), Eric D. Burry (Scientific Instrument Services, Ringoes, NJ), and Dr. Chad Quinn (SmithKline Beecham Pharmaceuticals, King of Prussia, PA).

The main goals of the chemical analyses were to identify any ancient organic components that might shed light on whether they represented “leftovers” of a funerary feast held before the burial, as proposed by Dr. Elizabeth Simpson, and to refine MASCA analytical methods.

Three independent chemical techniques are typically used by the MASCA laboratory to detect organic compounds, as outlined below. Samples were obtained by grinding up the solid ancient residue and extracting it twice with boiling methanol or chloroform for 20 minutes each.

1) Diffuse-Reflectance Infrared Fourier-Transform Spectrometry (DRIFTS) takes advantage of the nature of chemical bonds to stretch and bend when they absorb infrared (IR) light. Each chemical compound absorbs IR light at specific frequencies which can be precisely measured and shown on a spectrum. The technique is extremely versatile and precise, requiring as little as a milligram of material, which is ground up and mixed with potassium bromide, a transparent solid. The MASCA laboratory currently employs a Nicolet 5 DXB FT-IR spectrometer, with OMNIC 3.0 software and search capabilities. Spectra were desorbed at 8 cm⁻¹ wavenumber, a frequency unit used by spectroscopists for library storage, searches, and printing. Because the whole sample is analyzed simultaneously, the absorption peaks of individual compounds often overlap, sometimes frustrating accurate identifications.

2) High-Performance Liquid Chromatography (HPLC) is used for more precise identifications of mixed materials. Microgram amounts can be detected by MASCA's Hewlett-Packard 1090 Liquid Chromatograph, with an A06.01 ChemStation, which is run at 80 atm (ca. 1200 lbs/in²). A 10 or 20:1 sample, dissolved in methanol (or acetonitrile) is passed at 2 ml/min through a column (25 cm in length and 4.6 mm in diameter), which is lined with 3–10 micron diameter particles that preferentially absorb the compounds of interest. Depending on how strong the affinity or polarity is between the compound, moving solvent, and stationary substrate, the compound will take more or less time to pass through the

column (referred to as the retention time). Once separated, the components are fed into an ultraviolet (UV)-visible spectrophotometer diode array, ideally yielding characteristic absorptions of chromophores by the compounds of interest. A database of several hundred relevant archaeological samples and modern reference compounds is then searched by the ChemStation software for the best matches at a specific retention time and UV wavelength (usually 210 nm).

3) Feigl chemical spot tests, with microgram sensitivity, are used to test for specific compounds. For example, the presence of tartaric acid/tartrate, which occurs in large amounts naturally in the Middle East only in grapes (and therefore in wine), is confirmed by dissolving and heating an ancient sample in concentrated sulfuric acid. β, β’-dinitrophenol is then added, to convert tartaric acid to a compound that exhibits green fluorescence under UV light. Calcium oxalate or “beerstone,” which settles out at the bottom and along the sides of barley beer processing and storage vessels, is detected by reducing the sample with zinc granules in an acidic medium to glyoxallic acid, followed by reaction with phenylhydrazine and hydrogen peroxide, to give a distinctive pinkish red color.

In summary, the MASCA Molecular Archaeology Laboratory relies on three chemical techniques—DRIFTS, HPLC, and Feigl spot tests—to test an ancient organic sample for the presence of marker or fingerprint compounds, which are correlated to natural products of archaeological significance. A compound is said to be “present” only if it is attested by all three analyses. Follow-up liquid chromatographic-mass spectrometric (LC-MS) and gas chromatographic-mass spectrometric (GC-MS) analyses of some samples are useful in substantiating the findings, as well as in detecting additional compounds. Any assessment of the original organic material is also crucially dependent on and should be consistent with the archaeological context and the vessel types that contained the residues.

I. The “Mixed Beverage”: Wine, Barley Beer, and Honey Mead

Sixteen residues were tested from a range of bronze vessel types from Tumulus MM, which had contained more than 150 metal vessels, the most comprehensive Iron Age drinking set ever found, according to Moorey. Based on banqueting protocol at a royal celebration—depicted on wall reliefs in the palace of the Assyrian king Sargon II at Khorsabad, which dates to about the same time as the burial in Tumulus MM—a preliminary judgment about how each vessel was used in a “funerary banquet” could be hypothesized. The ram-headed and lion-headed “buckets” or situlae, for example, would have been used to transfer the beverage from the three large vats (or cauldrons) to smaller cauldrons, mounted on two special wooden serving stands. From there, the beverage would have been ladled into 98 bronze omphalos bowls and 19 large two-handled bowls. This hypothesis was confirmed by the analyses undertaken.

As catalogued below, the residues from one small cauldron, two situlae, seven omphalos bowls, and six other bowls were analyzed in the MASCA laboratory. This represented 57% (of a total of 28) of the samples from “beverage vessels” which were brought back to the University of Pennsylvania Museum following the 1957 expedition. Any residue originally present in the three large cauldrons had been destroyed by immersion in hot sodium hydroxide when the vessels were conserved.

All the “beverage” residues have a very similar macroscopic and microscopic physical appearance. They are intensely yellowish in color, with dark-colored platelets dispersed through the matrix. These platelets are very shiny and rippled on one surface, and matte-textured on the other, suggesting a liquid that originally evaporated along the inside of a vessel and then flaked off. At 80x magnification, numerous bubbles and particles (on the order of a half to 2 microns in diameter) are visible in the platelets.

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2 Feigl, Spots Tests in Organic Analysis.
3 See Young, Gordon I, for illustrations and discussion; the previous chemical analyses by A. Eric Parkinson are also included in this volume as an appendix. Gordon I, 277–284.
4 Moorey, “Metal Wine Sets,” 195.
List of Analyzed “Beverage” Samples

**MM101:** bronze small cauldron; analyzed by DRIFTS, HPLC, and chemical spot test for oxalate.

**MM45:** bronze lion-headed situla; analyzed by DRIFTS, HPLC, thermal desorption (TD)-GC-MS (Eric Butrym), and chemical spot tests for tartrate and oxalate.

**MM46:** bronze ram-headed situla; analyzed by DRIFTS, HPLC, LC-MS (Chad Quinn), GC-MS (Curt Beck and Edith Stout), and chemical spot tests for tartrate and oxalate.

**MM53:** bronze bowl with lifting handles; analyzed by DRIFTS, HPLC, and chemical spot tests for tartrate and oxalate.

**MM60:** bronze bowl with lifting handles; analyzed by DRIFTS, HPLC, and chemical spot test for oxalate.

**MM62:** bronze bowl with swiveling ring handles; analyzed by DRIFTS, HPLC, and chemical spot test for oxalate.

**MM66a:** bronze bowl with swiveling ring handles; analyzed by DRIFTS, HPLC, GC-MS (Robert Moreau and Alberto Nuñez), and chemical spot test for oxalate.

**MM66b:** bronze petaled omphalos bowl; analyzed by DRIFTS, HPLC, and chemical spot tests for tartrate and oxalate.

**MM65:** bronze petaled omphalos bowl; analyzed by DRIFTS, HPLC, and chemical spot test for tartrate.

**MM104:** bronze petaled omphalos bowl; analyzed by DRIFTS, HPLC, and chemical spot test for tartrate.

**MM28:** bronze petaled omphalos bowl; analyzed by DRIFTS, HPLC, and chemical spot test for tartrate.

**MM26:** bronze ribbed omphalos bowl; analyzed by DRIFTS, HPLC, GC-MS (Robert Moreau and Alberto Nuñez), and chemical spot tests for tartrate and oxalate.

**MM37:** bronze plain omphalos bowl; analyzed by DRIFTS, HPLC, and chemical spot tests for tartrate and oxalate.

**MM44:** bronze plain omphalos bowl; analyzed by DRIFTS, HPLC, and chemical spot test for tartrate.

**MM68:** bronze deep bowl; analyzed by DRIFTS and HPLC.

**MM69:** bronze deep bowl; analyzed by DRIFTS, HPLC, and chemical spot test for oxalate.

The chemical results for each group of samples—“beverage” and “food” (below)—were highly consistent within each group, but were very different from one another. When IR or HPLC searches were run on any given sample, its best matches were other samples in the same group. Thus, both the “beverage” and “food” represented in the tomb were of highly homogeneous chemical compositions.

The group of IR spectra shown in Text Figure 1 can be used to illustrate this conclusion. All the spectra have the same absorptions (variations in intensity are due to sample size or the amount of a given component), even though they come from a range of “beverage vessels,” and contrast with those of the “food” (Text Figure 3). Starting with the higher frequencies on the left of Text Figure 1, the broad band centered around 3400 cm\(^{-1}\) wavenumber is due to hydroxyl or water of hydration, and peaks at 2930 and 2860 cm\(^{-1}\) are characteristic of the stretching between carbon and hydrogen atoms (i.e., hydrocarbons); then comes the carbonyl band of various organic acids and esters at 1730–1710 cm\(^{-1}\), followed by carboxylate or organic acid salt bands between 1670 and 1570 cm\(^{-1}\). C–H bending frequencies show up at 1460, 1360, and 720 cm\(^{-1}\), along with numerous other matches between the samples in the so-called “fingerprint region” between 1300 and 600 cm\(^{-1}\).

By contrast, the “food” residues (Text Figure 3) are lacking the hydroxyl/water of hydration band around 3400 cm\(^{-1}\). The hydrocarbon C–H stretch bands are there, as in the “beverage” samples, but a slight shoulder at 3000 cm\(^{-1}\) sets them apart. A carbonyl band, which is often associated with organic acids and esters, is quite prominent, but it is much better defined in the “food,” with a greater intensity and at a higher frequency (1750–1730 cm\(^{-1}\)) than that of the “beverage.” Moreover, the broad carboxylate salt band (1670–1570 cm\(^{-1}\)), related to calcium tartrate and oxalate in the “beverage,” is just barely present. At lower frequencies, C–H bending shows up at 1470, 1420, and 1390 cm\(^{-1}\), and C–C stretching or C–H bending at 1170, 1120, and 720 cm\(^{-1}\), as was also noted for the “beverage.” But the fine structure of the “fingerprint region”—the shape, multiplicity, and intensity of the absorptions, representing any number of atomic vibrations—are very distinct for the “food” samples and quite unlike those of the “beverage” samples.
Text Figure 1. DRIFTS spectra showing the principal absorptions of Tumulus MM beverage samples from different vessel types. The MMto spectrum was less intense due to a smaller amount of material, accounting for its somewhat different appearance. See text for full explanation.
Text Figure 2. DRIFTS spectra of representative beverage sample from Tumulus MM, showing the principal absorptions as explained by synthetic calcium oxalate, calcium tartrate, and modern beeswax.
The IR results for the "beverage" samples can be explained as a mixture of grape, barley, and honey, most likely a combined fermented beverage of wine, beer, and mead, since naturally occurring yeast on grape skins and in honey become active in a liquid. Tartaric acid and tartrate salts are the fingerprint compounds of a grape product, because these compounds occur in large amounts naturally in the Mediterranean-Near Eastern region only in grape (Vitis vinifera). Outside of the Middle East and Europe, tartaric acid is also found in the African baobab tree and the fruit of the South Asian tamarind tree, but it is highly unlikely that products of these trees were traded during the early first millennium B.C. The insoluble potassium bitartrate and calcium tartrate salts are readily formed from the acid, and in unfiltered, unrefined wine, a crystalline accumulation of these salts, the so-called dregs or lees, will sometimes form on the bottom of a wine vessel. In Text Figure 1, tartaric acid is well attested by a sharp, intense carbonyl peak at 1720/1740 cm⁻¹, together with other absorptions at 1440 and 1250 cm⁻¹. Tartrate is es-
pecially pronounced in the samples, correlating with carboxylate absorptions at maxima of 1630 and 1560 cm\(^{-1}\), with additional peaks at 1550, 1430, 1380, 1320, 1270, 600, 560, and 480 cm\(^{-1}\) (cf. synthetic calcium tartrate in Text Figure 2). For all the samples tested, the presence of tartaric acid/tartrate was further borne out by positive Feigl spot tests and close HPLC matches with these compounds and other ancient and modern wine samples.

Calcium oxalate or beerstone, the marker compound for barley beer,\(^5\) accounts for the organic acid absorption at the higher end of the salt bands, between 1670 and 1610 cm\(^{-1}\). This compound also accounts for the carbonyl stretch absorptions at 1505 and 1330 cm\(^{-1}\) and some of the fine structure in the “fingerprint region” (cf. synthetic calcium oxalate, Text Figure 2). Positive Feigl spot tests for oxalate were obtained for all samples tested. HPLC gave close matches with the synthetic compound, as well as an ancient “ale” sample from New Kingdom Malkata\(^6\) and a “mixed beverage” from Late Bronze Age Greece (see Conclusions).

While tartaric acid/tartrate and calcium oxalate contribute minimally to the hydrocarbon stretch bands at 2930/2860 cm\(^{-1}\) (Text Figure 2), these peaks and a strong long-chain C-H bend absorption at around 725 cm\(^{-1}\) substantiate the presence of beeswax. Greater complexity in the carbonyl region below 1710 cm\(^{-1}\), coupled with doublet carbonyl peaks at 1730 and 1710 cm\(^{-1}\) due to ester bonds in the wax compounds, provide further evidence for this identification. Beeswax—with characteristic long hydrocarbons (especially the C\(_7\) compound, heptacosane) and related acids (in particular, the C\(_9\) compound, lignoceric acid)—provides an excellent group of fingerprint compounds for honey. Although the sugars in honey rapidly degrade, beeswax is virtually impossible to filter out completely when processing honey; and its compounds can be very well preserved.\(^7\) Curt Beck and Edith Stout identified the key compounds in beeswax by GC-MS in the samples that they analyzed. Close HPLC matches included beeswax, honey, mead (i.e., fermented honey beverage), potassium gluconate (the principal organic acid in honey), and “mixed beverages” with honey as one of the constituents from Mycenaean Greece and Minoan Crete. A starting material of honey, when diluted down to a third by water, will be fermented to mead by osmophilic yeasts in the honey.

In summary, the “beverage” samples from Tumulus MM are all chemically consistent with one another, and are best interpreted as a “mixed fermented beverage” of wine, barley beer, and honey mead. Their uniform chemical composition in the various “beverage” vessels implies that the three components had been mixed or prepared according to a fixed formula.

II. The “Food”: A Lamb or Goat Stew

The “food” residues were contained in 18 pottery vessels, six each inside the three large cauldrons, together with large clumps of similar-looking material randomly distributed in the cauldrons. If the large cauldrons were originally used to mix and serve the fermented beverage, then they must have been emptied before the “food” clumps and vessels were deposited in them.

Fourteen “food” residues, representing 54% (of a total of 26) of the samples brought back to Philadelphia, were tested. They included material from six dinos and four small amphorae (types described and illustrated in Young, Gordion I), and four clumps, as catalogued below.

Examination of the “food” residues revealed that they consisted uniformly of a brownish material, with a more fused, dark-colored surface, that was extremely contorted and with large depressed areas and holes running through it—quite unlike the thin, shiny platelets and intense yellowish matrix of the “beverage” (above). Tiny bubbles (2 microns in diameter) were seen at 80x magnification, but no cellular structures, seeds, grains, or other plant or animal materials were observed. The physical characteristics were in accord with a thick liquid that had congealed into a solid.

List of Analyzed “Food” Samples

MMI/36r: pottery dinos inside large cauldron MM 1; analyzed by DRIFTS, HPLC, LC-MS (Robert Moreau and Alberto Núñez; Chad Quinn), GC-MS (Curt Beck and Edith Stout),

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and chemical spot tests for tartrate and oxalate.

MM1/362: pottery dinos inside large cauldron MM 1; analyzed by DRIFTS, HPLC, LC-MS (Robert Moreau and Alberto Nuñez), GC-MS (Curt Beck and Edith Stout), and chemical spot test for tartrate.

MM1/372: pottery amphora inside large cauldron MM 1; analyzed by DRIFTS, HPLC, LC-MS (Robert Moreau and Alberto Nuñez), and TD-GC-MS (Eric Butrym).

MM1a: large clump inside large cauldron MM 1; analyzed by DRIFTS and HPLC.

MM1b: large clump inside large cauldron MM 1; analyzed by DRIFTS, HPLC, GC-MS (Curt Beck and Edith Stout), and chemical spot test for oxalate.

MM2/366: pottery dinos inside large cauldron MM 2; analyzed by DRIFTS and HPLC.

MM2/367: pottery dinos inside large cauldron MM 2; analyzed by DRIFTS, HPLC, LC-MS (Robert Moreau and Alberto Nuñez), TD-GC-MS (Eric Butrym), and chemical spot tests for tartrate and oxalate.

MM2/368: pottery dinos inside large cauldron MM 2; analyzed by DRIFTS, HPLC, and chemical spot test for oxalate.

MM2a: large clump inside large cauldron MM 2; analyzed by DRIFTS, HPLC, GC-MS (Curt Beck and Edith Stout).

MM3/370: pottery dinos inside large cauldron MM 3; analyzed by DRIFTS, HPLC, and GC-MS (Curt Beck and Edith Stout).

MM3/375: pottery amphora inside large cauldron MM 3; analyzed by DRIFTS, HPLC, GC-MS (Curt Beck and Edith Stout), and chemical spot test for tartrate.

MM3/376: pottery amphora inside large cauldron MM 3; analyzed by DRIFTS and HPLC.

MM3/377: pottery amphora inside large cauldron MM 3; analyzed by DRIFTS and HPLC.

MM3a: large clump inside large cauldron MM 3; analyzed by DRIFTS and HPLC.

As pointed out above, the “food” spectra shown in Text Figure 3 all have the same absorptions (although magnitudes may vary with relative amounts of each constituent and sample size), despite the fact that they come from different vessels.

The best reference matches in our IR database for the MM “food” residues, in addition to their matching one another, were modern lamb’s fat and beeswax (Text Figure 4). The slight shoulder at 3000 cm⁻¹ and a higher carbonyl absorption at 1755 cm⁻¹ set lamb’s fat apart from beeswax. Although the latter is present, marking the presence of honey, the overall congruity of lamb fat’s IR absorption with that of the MM “food” residues implies that it makes up the bulk of the composition.

The HPLC results told a similar story: besides being closely comparable to one another, the ancient “food” residues were most similar to modern lamb’s fat, honey, mead, potassium gluconate (the principal organic acid in honey), barley, calcium oxalate, and calcium tartrate. Relatively little absorption in the IR carboxylate region implied that tartrate, a marker for grapes or wine, was not a major constituent. The Feigl spot tests of the “food” samples confirmed the presence of tartaric acid/tartrate and oxalate.

The significant IR and HPLC matches between the ancient “food” samples and lamb’s fat are best accounted for by triglycerides, as confirmed by the LC-MS analyses of Robert Moreau and Alberto Nuñez. They showed that specific triglycerides were very prominent in the MM “food” remains, but not the “beverage” samples, viz., palmitodistearin (m/z 864, protonated), with lesser amounts of dipalmitostearin (836) and tripalmitin (808) (Text Figure 5). Small peaks at m/z 862, 892, and 890, respectively, are due to oleopalmito-stearin, tristearin and 2-oleodistearin, the latter being prevalent in pulses (i.e., the seeds of legumes). The diacylglycerides at m/z 608, 606, 580, and 552 are fragmentation products of the analytical process.

Other components of the “food” samples were identified by GC-MS and TD-GC-MS, carried out by Curt Beck, Edith Stout, and Eric Butrym. Their analyses showed that large quantities of the straight-chained C₆, C₈, C₁₀, C₁₂ saturated fatty acids—commonly known as caproic, caprylic, and capric acids, respectively—occur in the “food” but not the “beverage” residues. When these acids are found together, they are an excellent indicator of lamb or goat fat, as the Latin root (caper/capra meaning “goat”) suggests. There is no way to distinguish between the triglycerides or fatty acid composition of lamb and goat’s fat and meat, so the exact composition of the ancient “food” must be left open at present.

In addition to sheep or goat meat, a variety of other ingredients were detected in the “food” residues using GC-MS and TD-GC-MS. Phene-
Tumulus MM Dinos (MM1/361)

Lamb's fat

Beeswax

Text: Figure 4. DRIFTS spectra of representative beverage sample from Tumulus MM, showing the principal absorptions as explained by modern lamb’s fat and beeswax.

Anthrene, a stable aromatic hydrocarbon, and cresol, a phenol derivative, implied that the meat was first cooked over an open flame before it was cut off the bone. Honey, wine, and olive oil, which might have been used to marinate or barbecue the meat or to add their own distinctive flavors, were respectively represented by gluconic acid, tartaric acid, and oleic acid and its trans-isomer elaidic acid. Besides large amounts of cholesterol, which would be expected in a meat dish, a high-protein pulse—most likely, lentils—was present, as revealed by a related plant steroid, chondrillasterol (and as already borne out by the triglyceride 2-oleodistearin, above).

Although some components of the ancient “food” might have been prepared separately or could have been added at different stages, the uniform chemical composition of the contents of ten pottery vessels and four clumps that were analyzed strongly suggests that the end-product was a homogeneous stew; otherwise, one must imagine the individual components being divided up and distributed equally to each vessel.

The finishing touches to this stew were pro-
vided by herbs and spices, according to the GCMS and TD-GC-MS evidence: anisic acid (characteristic of anise or fennel) and α-terpineol and terpenoids found in various spices were identified. The sources of the latter compounds are unknown; bitter vetch (Vicia) and wild fenugreek (Trigonella), which grow around Gordian today, are possibilities, according to Dr. Naomi F. Miller of MASCA.

**Conclusions**

The chemical analyses of the “food” and “beverage” residues in Tumulus MM successfully identified the components of the hypothesized “funerary feast,” whose “leftovers” were deposited with the deceased after the funeral. The main entrée was a spicy barbecued lamb or goat stew with lentils and other vegetables (including barley). Noteworthy are the large stocks of lentils and cereals that were found in storage jars in the kitchens of buildings adjacent to what may have been Midas’s palace on the Gordion citadel.8

The collection of bronze vessels used to serve the “mixed fermented beverage” of wine, beer, and mead has an importance that extends well beyond a Phrygian funeral feast. Later Greeks would have turned up their noses at such a concoction, but Homer describes a drink (Greek ἱκέαμ) that combines wine, barley meal, honey, and goat’s cheese (Iliad 11:628–643; Odyssey 10:229–243). Ἦκεαμ is probably best translated as “mixture,” and a range of ancient Greek texts, extending down to Plato and the Eleusinian mysteries, suggests that any number of ingredients (herbs, spice, wine, milk, honey, oil, and water) might be tossed into the brew. Pliny the Elder

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(Natural History 14.113) claims that the best mead in the world was still being made in Phrygia in the first century A.D.

Recent chemical analyses by MASCA’s Molecular Archaeology Laboratory of numerous late Mycenaean and Minoan drinking vessels, dated between 1400 and 1350 B.C., have shown that they were filled with a mixed fermented beverage nearly identical to that from Tumulus MM. In other words, centuries before the Phrygians arrived in Anatolia, such a beverage was known and enjoyed in Greece and on Crete.

The main entrée of the funerary banquet also provided a strange, evocative parallel to Bronze Age Greek cuisine. As our evidence showed, a stew of sheep/goat, lentils, and other vegetables was eaten at the funerary feast associated with Tumulus MM. Similarly, lamb and lentil concoctions, sometimes flavored with olive oil, honey, wine, and various spices, figure prominently in Minoan and Mycenaean cuisines.

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