

MICROSCOPIC AND CHROMATOGRAPHIC ANALYSES
OF DECORATIVE BAND COLORS ON NABATEAN 'EN RAḤEL
TEXTILES—KERMES AND SHADED BANDS

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'En Raḥel, a Nabatean fort in the 'Arava Valley, was probably a way-station along the Spice Route from Petra to Gaza. It produced about 300 textile, basketry, cordage and related items (Shamir, this volume).

This article presents the results of the microscopic and chromatographic investigations of the colors of decorative bands found on some of the 2,000 year-old 'En Raḥel textiles from the Roman period (Table 1). The colors include various shades of orange, red, purple (reddish), blue and violet (bluish-purple). Optical microscopy was used to investigate the construction of the color of the examined yarn and the method used in the process of shading from one color to another in adjoining bands. Chemical dye analyses were performed by means of High-Performance Liquid Chromatography (HPLC), which can identify various dye components in a natural dyeing.

About 60 fiber samples from 'En Raḥel textiles were also analyzed via optical microscopy in order to identify the vegetal and animal sources of these fibers; these results were incorporated in the main report. Most of the yarns investigated consisted wholly of wool, and to a lesser degree of coarse goat hair, linen and camel hair. It was found that camel hair was not spun alone, but with goat hair, while yarns consisting of wool and goat hairs were formed by plying individual wool and goat-hair threads, and not by spinning with these two fibers.

Fleece Dyeing and Tri-Fleece Spinning

Table 1 summarizes the results of the microscopic analyses of the colors of the fibers in the yarns from various bands. One revealing obser-

vation from these investigations is that the colors in a number of the yarns were produced by spinning together three differently dyed woolen fleeces. Thus, each of the violet yarns in the bands from textiles Nos. 15 (2 samples; see Table 1) and 16 was produced from three fleeces, each of a different color. One fleece was dyed blue only, another red only, and the dominant purple fleece was double-dyed red and blue. The color in a few yarns from the brownish-orange band of No. 25 was obtained by spinning together two differently dyed fleeces, a relatively large amount of a reddish-orange fleece mixed with a bit of a crimson (bluish-red) fleece. (This same crimson fleece, by itself, was spun into yarn that comprised the purplish band from that textile.) The violet yarns of Nos. 15a and 16 are similar in color, and in each of these two yarns a predominant amount of a purple-dyed fleece was mixed with a bit of a blue fleece and an even lesser amount of a red fleece. In No. 15b, the violet coloration was produced by using approximately even amounts of a purple fleece and a blue-only fleece and a much smaller quantity of a red fleece.

The possibility that the red fibers present in these yarns were inadvertently included by the spinner—perhaps as a 'contaminant' from another fleece—and that they were not a deliberate addition to the yarn can be dismissed: the blue yarns from Nos. 15b and 16 consist of fibers that have only one color—blue. In No. 16, the absence of any spurious colored fibers from to-

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Table 1. Microscopic and Chromatographic Color Analyses of Woolen Decorations

Textile No.	Field Number		Fragments Examined	Color of Yarn	Decoration Type	Microscopic Color Analysis of Individual Fibers in Yarn	Dyes ^b Detected in Yarn via HPLC	Dyestuff Source of Fleece-Dyed Fibers
	Locus	Basket						
15 (a) [#]	13	134/19a	yarn	violet	band	purples > blues > reds	pu >> al; ind	purples = wild madder + indigotin ^c ; blues = indigotin reds = madder
15 (b) [#]	13	134/19b	yarn	blue	band	all fibers are blue	ind	indigotin
			yarn	violet	band	purples ~ blues >> reds	pu > al; ind	(similar to No. 15[a])
16 [*]	13	134/20	adjoining bands	blue	band	shading: ^a blues + blues >> reds ~ purples.	ind	indigotin
				violet	band	shading: ^a purples > blues > reds	pu >> al; ind	(same as No. 15[a])
21 [*]	13	169/3	yarn	red	tapestry	orange, red-orange	al > pu	madder
25 (a) ^{#*}	15	?/4	adjoining bands	purplish	band	pink-purple, crimson.	al >> pu	madder + Fe
				brownish orange	band	shading: ^a oranges + pinkish-orange >> crimson	al >> pu	orange = madder crimson = madder + Fe
25 (b) [#]	15	?/4b	yarn	orange	band	yellow-orange	al > pu	madder
25 (c) [#]	15	?/4c	yarn	orange-red	band	orange	al >> pu	madder
26a	15	?/5	yarn	purple	gamma	pink-lavender >> blue spots	ka >> ind	kermes + trace of indigotin
31 [*]	15	184/15	yarn	purple	band	pink, pink-purple	ka	kermes
32	15	184/16	yarn	purple	band	lavender, pink	ka	kermes
34 [*]	15	210/3	yarn	purple	band	lavender >> blue spots	ka >> ind	kermes + trace of indigotin
52	20	163/7	yarn	purple-red	band	pink	pu >> al; ind	wild madder + indigotin

[#] Multiple samples from a single textile.

^{*} See Color Plate.

^a See text.

^b pu = purpurin, al = alizarin, ind = indigotin, ka = kermesic acid.

^c "Indigotin" indicates an indigotin-producing plant, which may be either the woad or the indigo plant.

tally blue yarns is particularly important as these blue yarns form a band that directly neighbors the violet band. In addition, the red fibers, which appear in the three violet yarns mentioned above, cannot simply be the result of a poorly

double-dyed purple fleece because, if so, some blue would have been visible at the microscopic level. Each of the red fibers in the three violet yarns was completely monochromatic.

The Intricate Construction of the Shaded Bands

The morphologies of the two shaded bands (Nos. 16 and 25) were investigated and the results for the violet-blue sample (No. 16) are presented in Fig. 1. Most of the violet band is composed of nearly identical yarns formed from spinning together three different fleeces, as described above. Each of these main violet yarns was produced by using a large amount of a purple (red and blue double-dyed) fleece and successively smaller amounts of the blue and red fleeces. The other—blue—side of the band mostly consists of yarns from a blue-only fleece. The shading from one color to another was produced by means of color gradation on either side of the border. As can be seen from Fig. 1, on the violet side, four rows at the border have less purple and more blue than the main violet color. These yarns were also produced by spinning three differently dyed fleeces, but this time, about equal quantities of purple and blue fleeces were used. These four rows of yarn have nearly the same coloration. Their counterparts on the

other side of the border also consist of four rows of a single color, but this time only of blue. In order to temper this color transition from violet to blue, the weaver then introduced three rows of a mostly blue, but ever so slightly purplish, coloration. (Again, three different fleeces were used to produce these latter yarns.) In this way, the weaver created a gradual crossover from violet (bluish-purple) to blue according to the following coloring scheme:

BLUISH-PURPLE \Rightarrow (purple-blue) \Rightarrow (blue) \Rightarrow (purplish-blue) \Rightarrow BLUE.

Hence, four different yarns were used for these two adjoining bands.

In the brownish-orange and purplish adjoining bands of No. 25, the border has degraded and is not clear, but it appears that two or more border rows in the orange part have some crimson fibers in the yarn. This indicates that two fleeces were used to construct this yarn, employing a large amount of orange fleece and some crimson fleece. The shading in this textile is not as intricate as in the former case.

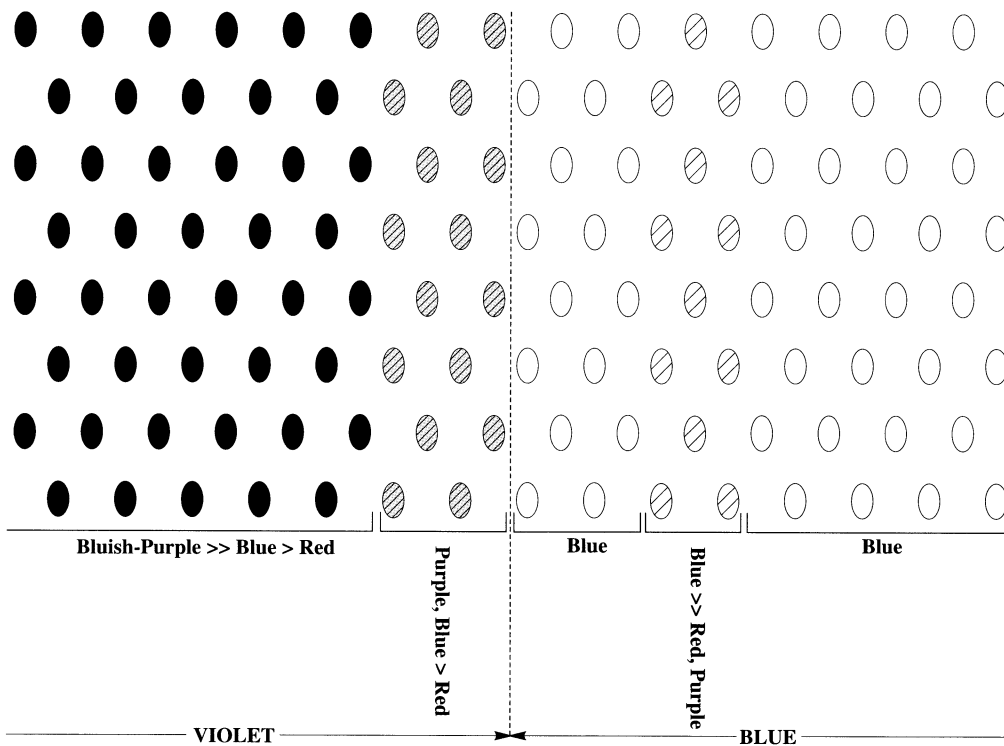


Fig. 1. The color morphology of the adjoining blue and violet bands from textile No. 16.

The Dyes

The dyes that were chemically extracted from these dyeings were chromatographically and spectrophotometrically analyzed via the reverse-phase HPLC technique (described in Koren 1994a; 1995). The instrumental setup used was newer and more sensitive than past dye analyses performed in this laboratory and consisted of a Waters™ 600E-model pump and 996-model photodiode array (PDA) detector. This system allowed the measurement of (a) the retention time, t_R , of each dye eluting out of the separation column and (b) its ultraviolet/visible absorption spectrum (absorbance units, AU, vs. wavelength). With the elution scheme and calibration method utilized in this study and described elsewhere (Koren 1994b), these two properties are, in general, sufficient for the positive identification of each main dye detected. Prior to the HPLC analysis, mordant and vat dyes need to be chemically extracted from the textile fibers by means of an appropriate solvent system (Koren 1994b). The results of these HPLC tests for each yarn examined are listed in Table 1.

The Wild Madder Plant, Regular Madder and Indigotin.— The blue dye, indigotin, identified in some of the dyeings, could have been obtained from the leaves of two plants, woad (*Isatis tinctoria* L.) or indigo (*Indigofera tinctoria*; Koren 1993). In Roman times, woad was cultivated in the Middle East (Koren 1996), and the indigo plant, originating in India and its environs—or the dye produced from it—could have been imported into the region by Nabatean traders and merchants.

The red-only dyeings that each yielded more alizarin (orange) than purpurin (purple) were produced using madder, *Rubia tinctorum* L., from the roots of which can be obtained these two major components (Koren 1993). However, the red dyestuff that was used, together with blue indigotin in a double-dyeing, to produce all the purple or violet hues, contained more purpurin than alizarin. This phenomenon is known for Roman-period purple or violet textiles, as, for example at Masada (Koren 1994a) and the Cave

of Letters (Koren, n.d). The plant species known as “wild madder” (*Rubia peregrina* L.), which was cultivated in the Levant, was used in these double dyeings. In laboratory tests of a modern dyeing produced by using the roots of a species of wild madder, purpurin was the major dye component found and no alizarin was detected (Wouters 1985).

The decision to use this purpurin-rich madder together with indigotin to produce purple colors was a logical technical choice for the dyer, who must have known that to obtain a quality “fake” of the real murex-purple, a madder that already contained a significant quantity of a purple colorant should be used. Modern dyeings performed by the author have shown that aluminum-mordanted purpurin-only dyeings on wool produce strawberry-red colors, whereas similar alizarin-only dyeings yield orange hues.

Though analyses of modern dyeings by a wild madder species did not detect any alizarin, the presence of alizarin in all the purpurin-rich samples in the current study can be explained by at least one of the following reasons: (1) The dyer of the ‘En Raḥel purples may have used a subspecies of *peregrina* that contains some alizarin. (2) The dyeings with wild madder were performed in the same vessel as other madder dyeings, which contain alizarin. As the residual dyes present in the vessel cannot be completely cleaned out of the vessel, since these dyes have only a limited solubility in water, the modern HPLC technique used in these dye analyses can detect quantities as low as even a nanogram (billionth of a gram). (3) Common alizarin-rich madder roots may have been added to wild madder in order (a) to obtain the desired shade, (b) to extend the wild madder, which was not as plentiful as regular madder, or (c) to dilute the more rare and expensive wild madder, so as to reduce cost and/or to deceive the buyer.

The presence of purpurin-rich madder roots together with indigotin for the production of “fake purple” in the ‘En Raḥel textiles can now be clearly established as a deliberate technique and not an inadvertent aspect of the dyeing process in the Roman era.

The Kermes Insect: Unprecedented Discovery.— The 'En Raḥel textiles provide the first evidence that the rare and expensive oak-kermes scale insect dye was used for ancient textiles found in Israel. Previous examinations of ancient textiles found in this region purportedly yielded only two textiles with the presence of an insect dye: the second century CE Bar-Kokhba purple ball of wool found at the Cave of Letters in the Judean Desert (Abrahams and Edelstein 1963; 1964) and the seventh-century CE 'En Boqeq red patch found near the Dead Sea (Masschelein-Kleiner et al. 1991). However, analyses performed by the author on these two textiles and on others from the same sites show that the Roman-period purple woolen fleeces were *not* dyed with an insect dye at all, but with wild madder and indigotin, and that the insect used in dyeing the Late Byzantine-period red textile could now be precisely identified as Armenian cochineal, a species growing on the upper roots of certain grassy weeds in the area of Mt. Ararat (Kurdian 1941; Donkin 1977; Verhecken and Wouters 1988/89).

Fig. 2 is a chromatogram of the purple dye extracted from 'En Raḥel textile No. 34. It shows the kermesic acid peak separated out of the column after about 10 minutes. This dye, not present in any plant, is found only in an oak-kermes species and not in a cochineal insect. The dried dark-brown pea-shaped *Kermes vermilio*, one of the most widely discussed scale-insect species (Donkin 1977; Verhecken and Wouters 1988/89; Cardon 1990a; 1990b), produces dyeings with two major dye components, flavokermesic (fk) acid and kermesic acid (ka); they can be detected by the HPLC method, as shown in Fig. 3. The fk dye is a yellow-orange solid that produces golden-yellow dyeings from acid baths (Mayer and Cook 1943:144), whereas solid ka is brick-red (Mayer and Cook 1943:141) or dark red (Merck 1989:833) and dyes wool orange-red from an acid bath (Mayer and Cook 1943:143). The ultraviolet and visible absorption spectrum of each of these two kermes dyes are depicted in Fig. 4. As both of these dyes are present in this *Kermes vermilio* species, alum-mordanted dyeings produced in this labo-

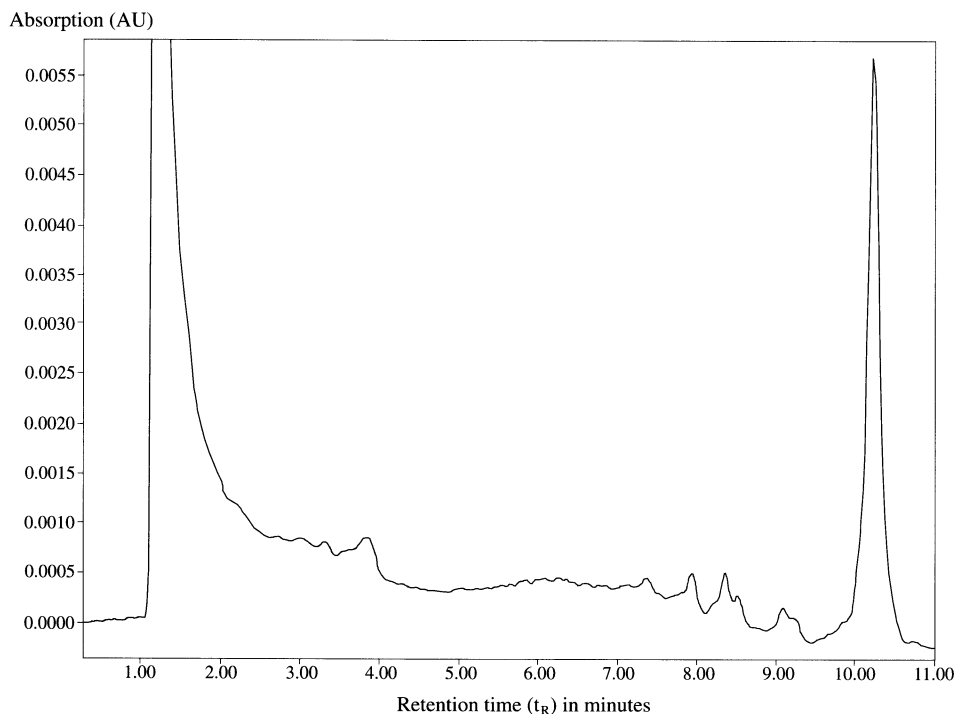


Fig. 2. HPLC chromatogram of the extracted dye solution from a purple yarn from the band of textile No. 34 indicating the kermesic acid peak eluting out of the separation column after about 10 minutes (with a detector wavelength of 275 nm).

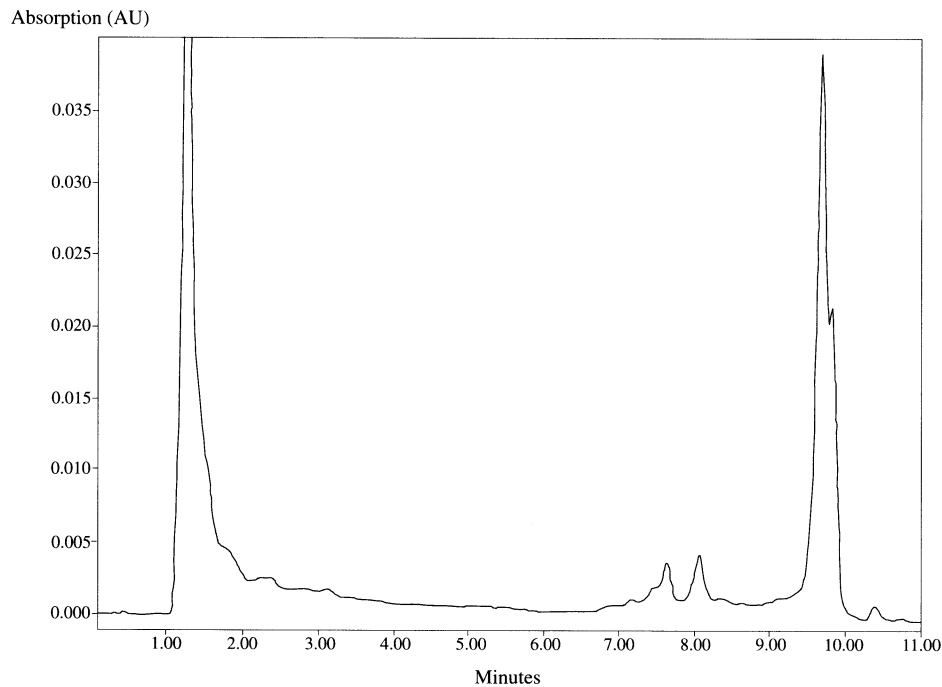


Fig. 3. HPLC chromatogram of the extracted dye solution from a modern *kermes vermilio* scale-insect dyeing indicating, after nearly 10 minutes, the 'kermes doublet' - flavokermesic acid closely followed by kermesic acid (with a detector wavelength of 275 nm).

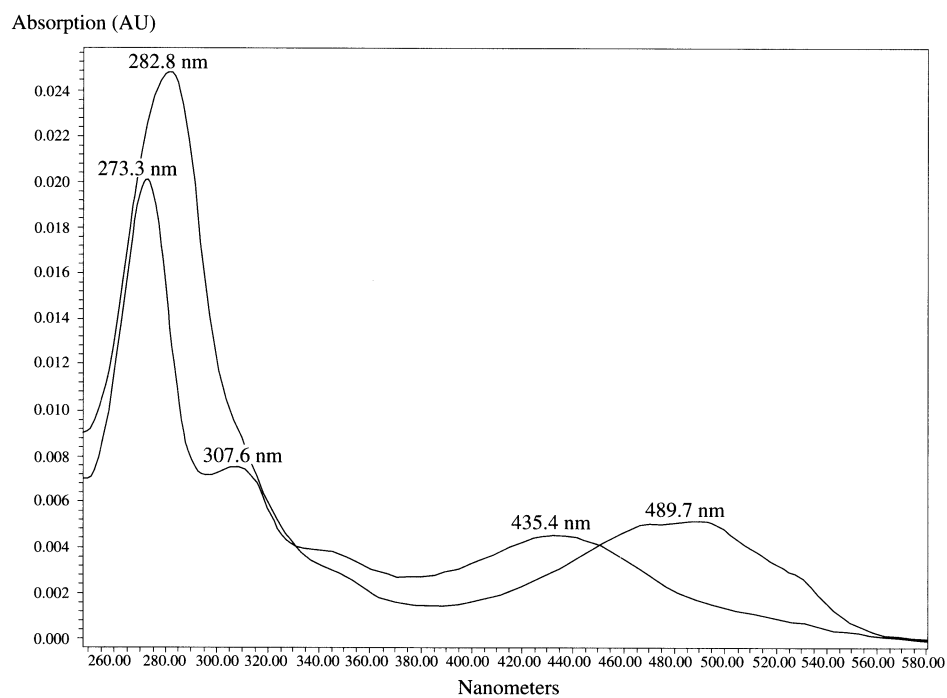


Fig. 4. Ultraviolet and visible absorption spectra of orange flavokermesic acid (wavelength of 435 nm at maximum visible absorbance) and crimson kermesic acid (comparable wavelength of 490 nm).

ratory with a modern species yielded orange-red (scarlet) colors.

In the analyses that were performed in this study and summarized in Table 1, four textiles were found to be dyed with a kermes insect, all from L15, the northwestern corner room (see Shamir, this volume: Plan 1). In two samples, kermes was the sole purple-producing dye, and in two others, kermes was mixed with a trace of indigotin to produce the desired purple color. However, only the crimson-colored ka dye was detected, and not the yellow-orange fk dye, which explains the redder (purplish) colors of the dyeings currently observed on the textiles. The absence of fk can be attributed to any one or more of the following factors: (a) a dyeing process or pre-dyeing process that the ancient dyer exploited to chemically enrich the dye bath with kermesic acid and to nearly totally eliminate fk from the solution (though this does not seem likely); (b) the use of a kermes species that is closely related to *vermilio*, which only contains ka; (c) degradation of the *vermilio* fk over the archaeological time span (some two millennia) elapsed since the deposition of the textiles.

Factors (b) and (c) are very important in that they have implications for the *original* color of the textile. The use of a kermes species that only contains ka means that the original color was similar to the present purplish hue. Alternatively, the plausible conjecture that the fk has degraded—yellowish dyes being less fast than reds or blues—would mean that the original color of that textile tended more to orange than is currently visible. The “aging factor” can be observed in Fig. 5, which incorporates data on the fk/ka ratios of adult female *vermilio* species from various circum-Mediterranean countries (Wouters 1990), as well as the relative fk and ka content in historic kermes dyeings (Wouters and Verhecken 1989). At 20% in a modern kermes dye, fk content consistently decreases as successively older kermes-dyed textiles are examined, until, as found in this study, 2,000 year old dyeings reveal no fk at all. Although more research is needed in this area to either affirm or negate this conjecture, the possible degradation of fk

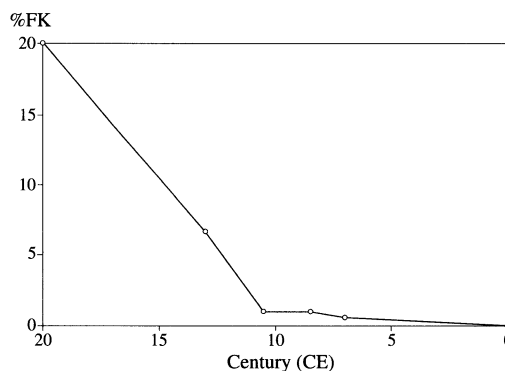


Fig. 5. Percent of the orange component (fk) of the kermes dye still present on historic textiles. The last data point, which refers to the “0th” century, is from this study; the others are from Wouters and Verhecken (1989).

over time is a factor that needs to be taken into account in interpreting the current analyses.

Summary—“Fake” Purples

The color and dye analyses have shown that the dyer, spinner and weaver were able to produce so-called “fake” purples and violets that mimicked the more expensive and more difficult to produce “real” colors from murex sea snails. These purples and violets were fashionable during the Roman period, especially for the elite of that society.

As observed in these ‘En Raḥel textile decorations (see Table 1) purples and violets were creatively produced by a variety of methods, which are outlined below:

By double-dyeing the fleece and then spinning into yarn:

Madder (regular) mordanted with an iron (Fe) salt

Wild madder + indigotin-plant

Kermes rich in kermesic acid

Kermes + indigotin-plant

By physically mixing differently colored fleeces in the spinning process, in the following ratios, to produce three differently colored yarns:

Purple > blue > red

Purple ~ blue > red

Blue >> red ~ purple

CONCLUSIONS

The analyses of the dyeings found at 'En Raḥel have revealed several important features regarding the color-production techniques in use in these Roman-period textiles. A major find of this study is the first bona-fide discovery of a kermes-insect dye in an ancient textile from Israel. Additional results pertain to the use of the wild madder plant, the various chemical and physical methods of creating purple and violet hues without the use of a murex snail dye, and the intricate shading process used in merging two adjoining bands. In general, the uniformity of dyeing in the samples analyzed is of a very

high quality; this homogeneity is even visible on the microscopic level. All these features attest to the high economic status of these Nabatean tradesmen and merchants, "sailors of the desert," living two thousand years ago.

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