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The Chemical Processing of Royal Purple Dye: Ancient Descriptions as Elucidated by Modern Science

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ABSTRACT Royal Purple, the most famous indigoid dye of antiquity, was derived from hypobranchial gland extracts of various marine gastropod mollusks. The extracts were processed by a lengthy, elaborate procedure, according to the Roman writer of the first century A.D., Pliny the Elder; other, more cursory texts are known from the Classical Greek period up until Byzantine times. In order to obtain fast, intense textile colors, as described in the ancient texts, a soluble precursor or derivative must have been used, which was then converted to the dye within the fiber. Present knowledge of the chemical compounds and reactions involved in indigoid dye processing and in the formation of Royal Purple from hypobranchial gland extracts suggests how this might have been accomplished by controlling the oxidative, photochemical, and/or enzymatic/hydrolytic pathways to the dye. Because of the imprecision and ambiguity of the ancient texts, however, the dyeing process can only be reconstructed by assuming that certain additives entered into specific reactions (e.g., a tin [not lead] reducing system in Pliny's account). Inferences are even more difficult to draw about the earliest known Royal Purple dye processing, based on the archaeological evidence from a thirteenth century B.C. industrial context at the site of Sarepta in Lebanon.

INTRODUCTION

Royal Purple (chemically known as 6,6′-dibromoindigotin) and related indigoids of molluscan origin are, as the most famous dyes of antiquity, frequently mentioned in ancient texts. One of the earliest references occurs in a letter (Knudtzon 1907–1914: I.162–163 [Letter no. 22]) which is part of the large corpus of international correspondence found at el-Amarna, the capital city of Akhenaten (1350–1334 B.C., following the lower chronology of Wente and Van Siclen 1976). The letter to Akhenaten's father, Amenhotep III, from Tushratta of Mitanni, an Indo-European kingdom on the upper Euphrates, mentions a shipment of violet-colored goods (Semitic *tekelet*, probably a mixture of dibromoindigo purple and indigo blue; see Ziderman 1981). About the same time, the dye is also referred to in the extensive literary corpus found at Ras Shamra (Thureau-Dangin 1934; Schaeffer 1950), ancient Ugarit, on the Syrian coast, as well as in Linear B texts from Crete (Stieglitz 1986: 183–184). Beyond the fact that Royal Purple, especially in the form of dyed fabrics, was an important commercial item and extensively traded, no information about the actual process of dyeing is provided in these texts.

In the succeeding centuries of the Iron Age, Canaanite city-states (Tyre, Sarepta, Sidon, etc.) on the Levantine coast came to dominate the dyeing industry (Bruin 1970; Pritchard 1978; McGovern and Michel 1984, 1985). Royal Purple was so intimately associated with these peoples that the name by which they called themselves (Canaanites), as well as their later historical appellation (Phoenicians), very likely derived from ancient Semitic and Greek roots for "purple" (Speiser 1936; Landsberger 1967). Perhaps as much as the Semitic alphabet or the seafaring by which the latter was transmitted to the West, purple dyeing was a distinctive element of Phoenician culture, and to promote the industry, dye factories were set up at colonies throughout the Mediterranean (Reese 1979–80). Given the importance attached to Royal Purple, it is somewhat surprising to find that the dye is rarely referred to in the known Phoenician texts, and that the process of dyeing itself goes totally unmentioned. Indeed, Biblical texts, which incorporate Iron Age traditions, are more informative about the involvement of Phoenician city-states, especially Tyre (Ezekiel 27:7, 16, 24; II Chronicles 2:7, 14), in the industry. The use of Royal Purple in early Israelite religion (e.g., in the tabernacle curtains and the High Priest's vestments [Exodus 26:1, 31; 28:4–6; 39:1, 28–29; etc.; II Chronicles 3:14]) shows considerable Phoenician influence, which was especially strong during the time of Solomon, when craftsmen from Phoenicia were employed to construct the Temple in Jerusalem, using imported Lebanese cedar and other materials (I Kings 5:1–12; 7:13–14; 9:10–14, 26–28; 10:11, 22). The apparent textual omission of Royal Purple could reflect the very limited archaeological investigation of Iron Age sites in Lebanon. In addition, industrial processes might have been held in low esteem by writers, or they might have been closely guarded secrets of the initiated; in either case they were not properly in the domain of written documentation. As is evident from the glassmaking texts of the early second millennium B.C. (Oppenheim et al. 1970), however, the manufacturing procedures of some ancient crafts could be described in great detail, with a highly specialized vocabulary.

Classical Greek sources also give little more than brief notations about Royal Purple. Aristotle (*Historia Animalium*, book V, chapters 12 and 15; *De Coloribus*, chapter 4) stated that the dye was derived from a specific organ of various marine gastropod mollusks, which had to be removed with extreme care from beneath the shell. The Greek legend about the discovery of the dye, which was repeated in various versions in later texts (see especially that of Julius Pollux of the second century A.D. [*Onomasticon* I, 45–49]), was that a dog belonging to Herakles, the god of Tyre, bit into a large sea-snail, staining its mouth red, whereupon Herakles promptly dyed a garment with the newly discovered substance. Beyond the fact that a red molluscan extract could be used to dye a textile by direct application, this story sheds no light on the processing of Royal Purple.

It is only in the time of the early Roman Empire, as much as 1500 years after the industry began, that the process is first described in some detail. The longest account is found in the *Historia Naturalis* of Pliny the Elder (book IX, sections 60–65, chapters XXXVI–XLI, as translated in the edition of Bailey 1929), written in the mid-first century A.D.:

> Two kinds of shell-fish furnish the purple and the conchylian dyes—the colours used for both are the same, but they are mixed in different proportions. . . .
>
> Purples are caught with a sort of small wicker basket cast into the deep, and containing as bait bivalves which snap their shells together, as mussels are known to do. These bivalves, though half-dead, revive on returning to the sea and gape open greedily. The purples seek them out and attack them with protruding tongue, but the mussels shut up as soon they feel the sting, and hold their assailants fast. Thus suspended, the purples are taken up, caught by their own greed.
>
> The best time to catch them is after the rising of the dog-star or before spring arrives, for, when they have produced the honeycomb-like exudation, the juice is too thin. Yet this fact, although of the utmost importance, is not recognised in the dye-factories. The vein already mentioned is then extracted and about a sextarius [ca. 7 lb.] of salt added to each hundred pounds of material. It should be soaked for three days, for the fresher the extract, the more powerful the dye, then boiled in a leaden vessel. Next, five hundred pounds of dye-stuff, diluted with an amphora [about 8 gallons] of water, are subject to an even and moderate heat by placing the vessels in a flue communicating with a distant furnace.
>
> Meanwhile the flesh which necessarily adheres to the veins is skimmed off and a test is made about the tenth day by steeping a well-washed fleece in the liquefied contents of one of the vessels. The liquid is then heated till the colour answers to expectations. A frankly red colour is inferior to one with a tinge of black. The wool drinks in the dye for five hours and after carding is dipped again and again until all the colour is absorbed.
>
> . . . The Tyrian colour is obtained by first steeping the wool in a raw and unheated vat of pelagian extract, and then transferring to one of buccine. . . .
>
> Conchyliated garments are prepared by a similar method to the first, but no buccine extract is used and the dye is diluted simultaneously with water and human urine. Only half the usual amount of dye is used. . . .

Considering the wealth of detail in Pliny's account, greatly exceeding that of any other contemporary document, he must have either observed the industrial process first-hand or received information about it from a knowledgeable source. In brief, he describes the capture of certain mollusk species, and the removal of a colorless organ from each specimen. These organs were then subjected to a sequence of operations with salt and water in vessels of lead (as translated, but see below). The mixture was then heated over a ten-day period, refuse organic materials were skimmed off the surface, and the liquid was tested for its dyeing properties. The use of varying amounts of extract from different mollusk species, as well as the dilution of the mixture with water and urine, suggests that the coloration could be changed by modifying the process.

Relatively little additional information about the Royal Purple dyeing process can be obtained from other Roman writings. In Plutarch's biography of Alexander, the fastness of the dye is attested by Alexander's recovery of 5000 talents of very well preserved purple textiles from the Persian court at Susa, which are said to have originated 190 years earlier at Hermione in Greece. A possibly significant note by Plutarch is that this cloth was dyed by using honey in some instances and oil in others (see also the commentary in Blümner 1942). The alchemical texts written in Egypt in the third century A.D., in particular Papyri Leidensis and Graecus Holmiensis (editions of Berthelot 1887 and Lagercrantz 1913, respectively), with probable roots in much earlier tradition (Reinking 1925; Forbes 1964), mention specific materials and procedures that entered into the processing of purple dyes. But since the goal of these experimenters was evidently to manufacture an economical substitute for Royal Purple, it is unclear whether the precursors of mollusk-, plant-, or insect-derived dyes are being described in a given passage. Talmudic descriptions of the same period or later refer to marine animal dyes, probably including Royal Purple, that were processed by heating the "blood" of the animal (most likely, the hypobranchial secretions) and tested in a mixture of urine, alumina, and fenugreek, or in fermented barley flour dough (Herzog 1919–20).

Following the Islamic conquest of the Middle East in the early seventh century A.D., Royal Purple production was greatly curtailed. Even Mediterranean dye works, unaffected by the invasions, ceased to operate, because of the increased availability of less expensive substitutes. Consequently, medieval references are primarily dependent on earlier writings. Production ceased altogether after the fall of Constantinople in A.D. 1453. In 1464, Pope Paul II issued an edict instructing cardinals to substitute cochineal scarlet for Royal Purple in dyeing their vestments (see Jensen 1963 and Born 1937).

In this brief historical overview of the written references to the processing of Royal Purple, Pliny's account stands out as the most lengthy and authoritative. Modern writers on the subject, like their ancient counterparts, consequently turned to it in attempting to reconstruct or describe the dyeing process. Since Pliny did not claim to give a complete account of the process, however, writers have felt justified in supplying additional details in accord with the scientific, historical, and linguistic data available to them. In the ongoing process of understanding Pliny's account, it is to be expected that sometimes the text was overly elaborated upon or that unwarranted inferences were drawn from it. In order that inquiry not be prejudiced by past interpretations, and to direct future research in the most fruitful directions, it is necessary to reappraise the evidence and its direct relevance to the ancient text from time to time. The remaining part of this article is such an interpretative effort from the perspective of modern science, first outlining the chemistry of Royal Purple, as currently known, and then suggesting ways in which specific chemical compounds and reactions might be related to Pliny's account and other more cursory or ambiguous ancient texts.

THE CHEMISTRY OF ROYAL PURPLE

As early as 1685, it was noted by Cole that a colorless fluid in the hypobranchial glands of marine mollusks found off the coast of Britain was converted to a red color on exposure to light. Subsequent research (Lacaze-Duthiers 1859) substantiated the photochemical nature of the process. During the nineteenth century, as one of the earliest chapters in the emerging field of archaeological chemistry, an attempt was made to identify the chemical structure of the dye (Negri 1875; Schunck 1879). Finally, in 1909, Friedländer determined that 6,6′-dibromoindigotin was the dye obtained from one of the Mediterranean mollusk species—*Murex brandaris*. That this compound was also a major component of the dyes from other Mediterranean species (*Murex trunculus* and *Purpura haemastoma*), as well as from mollusk species occurring in other parts of the world (e.g., *Purpura aperta* and *Nucella [Purpura] lapillus* from the Gulf of Mexico and the Atlantic Ocean, respectively), was supported by later research of Friedländer (1922) and others (Bouchilloux and Roche 1955; Fouquet and Bielig 1971).

The isolation and structures of the individual precursors were first effectively investigated by Bouchilloux and Roche (1955), and later more completely elucidated by Baker and Sutherland (1968) and Fouquet and Bielig (1971). The precursors were found to be sulfate esters of indoxyl, 6-bromoindoxyl, and derivatives of these substituted in the 2 position with methylthio or methyl sulfonyl groups (see compounds I and V in Fig. 1). Hydrolysis of these sulfate esters by the enzyme purpurase (step 1 in Fig. 1) was studied by Dubois (1909) and Erspamer (1947). Baker and Sutherland (1968) also showed that the oxidatively formed greenish compound from 2-substituted indoxyls converted to dye by a photochemical mechanism (step 2c in Fig. 1), in the process evolving long-observed, odoriferous sulfur compounds. The structure of this intermediate (compound VIII in Fig. 1) was identified by Christophersen and co-workers (1978) as a 2,2′-bissubstituted 2,2′-diindoxyl, the dibromo derivative being referred to as tyriverdin. Indoxyls not substituted in the 2 position were shown to form indigoids directly by oxidative coupling in air. Whereas *Murex trunculus* secretions contained both indoxyls and 6-bromoindoxyls, substituted and unsubstituted in the 2 position, extracts from *Murex brandaris* and *Purpura haemastoma* contained only 2-substituted 6-bromoindoxyl. Consequently, *Murex trunculus* yields both indigotin and 6,6′-dibromoindigotin, while *Purpura haemastoma* and *Murex brandaris* produce exclusively 6,6′-dibromoindigotin.

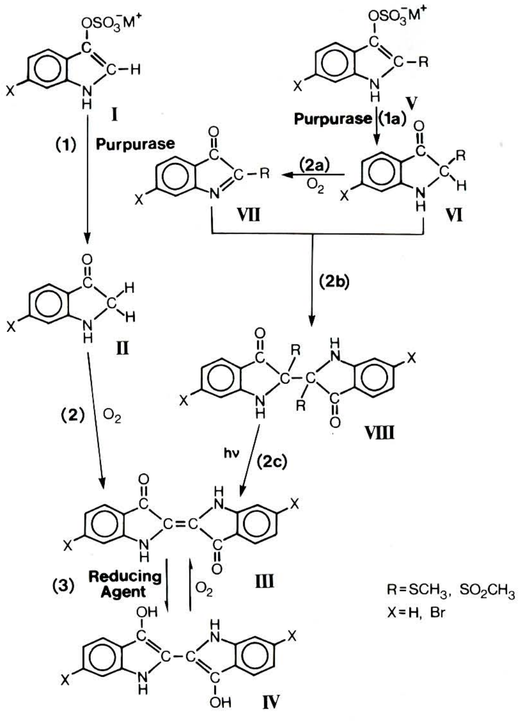

Fig. 1. Production of 6,6′-dibromoindigotin

As indigo vat dyeing is practiced today, the indigoid is reduced to the almost colorless, soluble leuco base (step 3, going from compound III to IV, in Fig. 1), in which form it is absorbed by the textile to be dyed. Reoxidation by exposure to air yields the colored dye, which is so well bonded to the textile that it is wash-fast and resistant to rubbing. This procedure is to be contrasted with direct application of the mollusk extract to a textile with subsequent color development in the sun, such as was

observed in the British Isles in the seventeenth century by Cole (1685), or in Mexico as still practiced by the Indians (Nuttal 1909; Gerhard 1977).

Any process which does not use the mollusk secretions directly must in some manner avoid the formation of the insoluble dye until the textile fiber has been impregnated. This might be accomplished in a number of ways:

1. The substituted or unsubstituted dye, once formed, could be converted to the leuco base by chemical or fermentative reduction (step 3 in Fig. 1). Even with excess reducing agent present, however, care would be needed to minimize exposure of the reduced solution to air.
2. For those mollusk extracts containing only 2-substituted indoxyls or for those that were obtained by separating the unsubstituted indoxyls after oxidative coupling (step 2 in Fig. 1), the avoidance of light would prevent the conversion of diindoxyls to indigoid dye (step 2c, going from compound VIII to III, in Fig. 1).
3. The formation of the diindoxyls from indoxyls (starting with step 2a in Fig. 1) might be blocked by antioxidants, i.e., stabilizers to oxidation of the intermediate (compound VI in Fig. 1).
4. The hydrolysis of the sulfate ester precursors might be blocked by the deactivation of the enzyme purpurase (steps 1 and 1a in Fig. 1).

Although the ancient dyer lacked an understanding of the chemistry of Royal Purple, the time-consuming, elaborate procedures detailed in Pliny and other sources go far beyond the simple application of the mollusk extract and probably have a basis in pragmatic observations on how best to produce an uncontaminated, fast, attractive textile dye. The task of the interpreter is to determine whether any of the above chemical methods aids in the understanding of an ancient text.

THE ANCIENT PROCESSING OF ROYAL PURPLE

Chemical and Fermentative Reduction Systems

The ancient extraction process for Royal Purple as described by Pliny has often been understood as a vat process (e.g., Heinisch 1957; Forbes 1964: 99–150; Bruin 1970; and Reese 1979–80; contrast Baker 1974). Actual experiments according to the recipes and procedures outlined by Pliny, however, are only able to produce a true vat dye under certain circumstances that may or may not conform to the intended meaning of the text. For example, the word *plumbum* can be translated as "tin" (Pliny the Elder 1855, vol. 2: 446, n. 93; Caley 1971) or "lead" (Bailey 1929), depending upon whether one supplies the adjective *album* ("white") or *nigrum* ("black"); neither appears in Pliny's text. The choice of adjective is critical in deciding whether Pliny is describing a reducing system. Lead and an alkaline solution will not reduce indigoids, but, according to experiments carried out in the MASCA laboratories, reduction to the leuco base occurs slowly with metallic tin in a potash solution at ca. 90°C.* Accordingly, the prolonged heating of the mollusk extracts in an alkaline solution in a tin or tin-coated vessel could have reduced the indigoid to the leuco base. Different colors could have been achieved by dipping the textile in the leuco base solutions from different mollusk species for varying periods of time. Pliny, however, does not explicitly state that an alkali was used, and the alkalinity of the additives he does mention (e.g., urine and salt) would not have been sufficient for reduction to occur. Possibly this was an oversight in his text, since strong alkalis (potash, soda, and lime) were available to the Romans (Forbes 1965: 181–188, 243).

An iron filing/fermented urine mixture at 90°C has been shown to be effective in our laboratory studies in reducing indigotin, but not 6,6′-dibromoindigotin. The purple dye processed with iron and urine, described in Papyrus Leidensis, must then not have been a mollusk purple. Furthermore, iron and urine form iron salts, and might have served as mordants for red dyes from madder or kermes.

It has been recently proposed by Elsner

*Similar experiments by Doumet (1980) at moderate temperatures (40°C) were unsuccessful in dissolving (= reducing) Royal Purple.

and Spanier (1985) that the 2-methylthio- and 2-methyl sulfonyl-substituted indoxyl dye precursors from the hypobranchial gland of the various mollusk species are the source of reducing agents for indigoid dyes, thus setting up a natural vat system. In the experiments of Elsner and Spanier, since only a finite amount of the 2-substituted indoxyls is available for reduction, reoxidation by the air is prevented by a surface layer of oil or the addition of a reducing sugar (glucose from grape juice). As noted above, evidence exists in ancient texts for the use of such materials in the processing of Royal Purple. For example, honey, mentioned by Plutarch, is a mixture of glucose and fructose, and has been shown to be an effective reducing agent for indigotin (although thus far undemonstrated for dibromoindigotin).

Honey and other organics might also have entered into a fermentative reduction process (cf. Jensen 1963). A very clear description of a fermentation vat in the processing of the woad plant, a source of indigo, is found in Papyrus Graecus Holmiensis. Presumably, mollusk dyers would have known about the same process. In the mollusk extraction, the animal residues could also have fermented, especially if large amounts of the materials were involved and the processing took place over several days. If the extract solution were boiled, as described by Pliny (see above), then fermentation would be halted and the solution sterilized; if temperatures were kept well below 100°C during the remainder of the process ("an even and moderate heat by placing the vessels [with the extract solution] in a flue communicating with a distant furnace"), then fermentation might have been reinitiated.

Some modern investigators (Heinisch 1957; Jensen 1963) conjecture that the marine lichen orseille acted as a reducing agent or a stabilizer for intermediates (see below). This has never been tested chemically, and the various ancient references (Theophrastus of Eresos, *De Historia Plantarum,* book IV, chapter 6, paragraph 5; Pliny the Elder, *Historia Naturalis,* book XXVI, section 103, chapter LXVI; Papyrus Graecus Holmiensis, in Lagercrantz 1913: *passim*) rather suggest that orseille, a reddish purple dye itself (a phenolic phenoxazine derivative used to make litmus), was a source of ground-color for the Royal Purple.

Photochemical Control

By processing the molluscan extracts in the dark or at least in subdued lighting (with lower levels of ultraviolet radiation), it might have been possible to block the photolysis of the diindoxyl (step 2c in Fig. 1). This procedure would have been only partially successful in preventing dye precipitation in species (viz., *Murex trunculus*) producing both 2-substituted and unsubstituted indoxyls, but completely so for species (*Murex brandaris* and *Purpura haemastoma*) that secrete only substituted indoxyls. Although ancient dyers must have observed that certain mollusk extracts developed a deeper color more quickly in sunlight than in the dark, surprisingly, no ancient text makes reference to the lighting conditions (whether total darkness, partial light, or total sunlight) for any stage of the process.

Indoxyl Stabilization

Besides preserving the dye in the leuco form (compound IV in Fig. 1), reducing agents might also have been used to block the oxidative pathways from the indoxyls to the indigoid (step 2, going from compound II to III in Fig. 1) or to the indolineone (step 2a, going from compound VI to VII). The work of Doumet (1980) indicates that the precipitation of the dye can be avoided during room-temperature extraction of the glandular materials if alkaline, aqueous media in vessels of lead alloyed with tin, antimony, or arsenic are utilized. During extract concentration at higher temperatures (40–50°C) and for longer periods, conditions similar to those described in Pliny's account, formation of the dye could not be prevented unless pure tin was used. On the other hand, starting with the dye, it could not be dissolved under the latter, more strenuous conditions. The implication of Doumet's experiments is that precursor oxidative pathways could be blocked under the appropriate conditions.

Deactivation of Purpurase

Elsner and Spanier (1985) have shown that the enzyme purpurase, which occurs naturally in the extract and is essential for the hydrolysis of the precursors (steps 1 and 1a in Fig. 1), can be deactivated by placing the freshly excised hypobranchial glands in 75°C water. An effective dyeing process might then have been to impregnate the textile with the deactivated precursor solution, and to add back purpurase to reinitiate the dyeing process. This suggestion, however, is not supported by the ancient textual evidence, the most complete account by Pliny indicating that the mollusks were in fact soaked in a cold brine before extraction.

CONCLUSIONS

By clearly distinguishing between the various chemical compounds and reactions involved in indigoid dye processing, the information provided in ancient texts, especially that of Pliny the Elder, can be assessed in terms of the dyeing procedures carried out in antiquity. Very often, the mention of a material (e.g., honey, tin or lead, orseille), even in the context of an industrial process, is not sufficient to establish the exact purpose of that material, whether as a chemical or fermentative reducing agent in a vat process, an additive to block oxidative pathways to the indigoid, a color additive, and so forth. The available ancient texts dealing with Royal Purple are probably too equivocal or imprecise in their vocabularies and descriptions ever to provide the sort of exactitude that can be meaningfully interrelated with results from modern scientific investigation. This is not to say that other texts might not eventually be found that will help in understanding better some of the stages in the ancient processing of Royal Purple. For example, a statement about the approximate temperature at which the concentration of the extracts was carried out would help in deciding whether a specific reducing system could have been operative. The role of darkness and sunlight in the ancient process is also not clearly specified in ancient texts, yet this is crucial to understanding whether some of the dye precursors could be kept in solution.

If problems exist in relating the descriptions of Pliny and other Roman and Greek writers to the known chemistry of Royal Purple, then extrapolating the Roman process back into Phoenician and earlier times is even more difficult. The most that can be done is to hypothesize what processes might have been employed on the basis of the archaeological data. We (1984, 1985) have previously discussed what is thus far the only documented instance of a dyeing facility for Royal Purple at Sarepta in the Phoenician homeland of Lebanon, dating to the thirteenth century B.C. (Pritchard 1980). A spouted vat with 6,6′-dibromoindigotin on its interior was interpreted as a processing container by which liquids could be drained off and solid organic residues scooped from the surface. Since the dyeing facility was in the midst of a large group of pottery kilns (Khalifeh in press), in which pottery was fired to a temperature at least above 500°C, possibly the fresh extract mixture was exposed to temperatures near 100°C, which would have deactivated the enzyme purpurase and aided reductive reactions under appropriate conditions. The apparent contradiction between the presence of only the dibromo compound on the interiors of pottery vessels and the fact that only broken shells of *Murex trunculus,* whose secretions contain a mixture of brominated and unbrominated precursors, were found in the vicinity of the dyeing installation led to another hypothesis. If the *Murex trunculus* extract solution were kept in darkness, the indoxyls not substituted in the 2 position, which form about 90 percent of the indigotin blue, could have been prematurely converted to the dye and then separated from the solution containing mostly the dibromo compound. None of the chemical reconstructions can be proven, yet they do provide a framework in which to carry out additional chemical and archaeological investigation that may eventually shed further light on the pre-Roman industry.

Much more still remains to be learned about the chemistry of the mollusk indigoid dyes. The reactivity of indigotin has been extensively studied in the laboratory, but that of dibromoindigotin, which can be expected to have significantly different reaction rates, is only partly known. More exact information on the effect of reducing systems, natural antioxidants, heat, and various additives (e.g., salts, acids, and bases) on the stability of the indoxyl precursors is also needed. Analyses of extracts from different species and sexes at various seasons of the year would be of value in determining the relative amounts of precursors, enzymes, and other substances that could affect the reaction. Still, even as the basis of speculation is improved by continued chemical experimentation, our understanding of the ancient processing of Royal Purple will always have an inherent limitation in the available textual evidence.

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