side" of the "ordered surface" free energy peak, and spontaneous growth of the droplet occurs. In effect, the transition size becomes the critical size for nucleation, and the free energy peak is ΔF_j ($j = j_t$), which is less than ΔF_j ($j = j_c$) for both models. This lower free energy peak results in an increase of 0.33×10^3 in the steady-state nucleation rate with respect to the rate for the state with a disordered surface (model 1) (*8*).

The features of the ΔF_j curves (Fig. 1) are dependent on the choice of T and S. For my choice of parameters, j_t is less than j_c. However, it is possible to choose the environmental conditions such that the converse is true (for example, $T = 232.2°K$, $S = 10$, $j_c = 79$ for the model with a disordered surface, and $j_t = 118$ or $j_t > j_c$).

Thus far in the discussion I have assumed that, when a droplet reaches the transition size j_t, it instantaneously relaxes to the state with an ordered surface. The orientation relaxation frequencies of polar molecules in the bulk liquid or in the solid state vary over a wide range and are strongly temperature-dependent. For water at room temperature, the relaxation frequency is about 3×10^{10} cycle sec^{-1} (*9*). In ice at −20°C the relaxation frequency is of the order of 10^3 cycle sec^{-1} (*9*). A comparison of this orientation relaxation time with the mean time needed for a droplet of j_t molecules to grow until it reaches j_c size will give an indication of whether the equilibrium argument is justified. White (*10*) has recently studied the kinetics of steady-state "random walks" on the positive integers and has applied the theory to nucleation phenomena. He finds that the mean time τ_{j_t} it takes a droplet of size j_t to traverse the range of sizes to the critical size is given by

$$\tau_{j_t} = \sum_{i = j_t}^{j_c} N_i / I \sim N_{j_t}{}^{e} I^{-1}$$

where N_i is the steady-state concentration of droplets of i molecules, I is the steady-state current, and $N_{j_t}{}^{e}$ is the equilibrium concentration of droplets of transition size j_t. For the example presented here, τ_{j_t} is $\sim 10^{-5}$ second. Depending on whether we picture the surface dipoles as relaxing like dipoles in room-temperature water or like dipoles in ice, we may conclude that the droplet grows in the state with an ordered surface for $j > j_t$ or that it follows the path of the free energy curve for the disordered surface to nucleation at $j_c =$ 150. Since most experiments are performed under conditions in which the embryonic droplets are in the supercooled water state, a state that is nearly approximated by the structure of ice, one would be tempted to conclude that droplets follow the path of the free energy curve for the disordered surface.

Although it is of fundamental physical interest to resolve this question, the difference of 10^3 in the steady-state current for the two models is not significant in a determination of the critical supersaturation. However, it is clear that a knowledge of absolute nucleation parameters must await a better knowledge of the fine structure of macromolecular systems (that is, $j \simeq$ 100 molecules).

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13 November 1969

Palmoxylon simperi and Palmoxylon pristina: Two Pre-Cretaceous Angiosperms from Utah

Abstract. *Petrified palm stems* (Palmoxylon simperi *and* Palmoxylon pristina) *from the Middle Jurassic Arapien Shale Formation near Redmond, Sevier County, Utah, are the first unequivocal angiosperms reported from pre-Cretaceous strata.*

The search for pre-Cretaceous angiosperms is an old one, and although many authors have proclaimed such a discovery, none of their claims has survived the critical investigations of subsequent researchers. The point of origin for the angiosperms is considered by some authors to be a sharp one (*1*) with the Lower Cretaceous being the boundary. It is estimated that

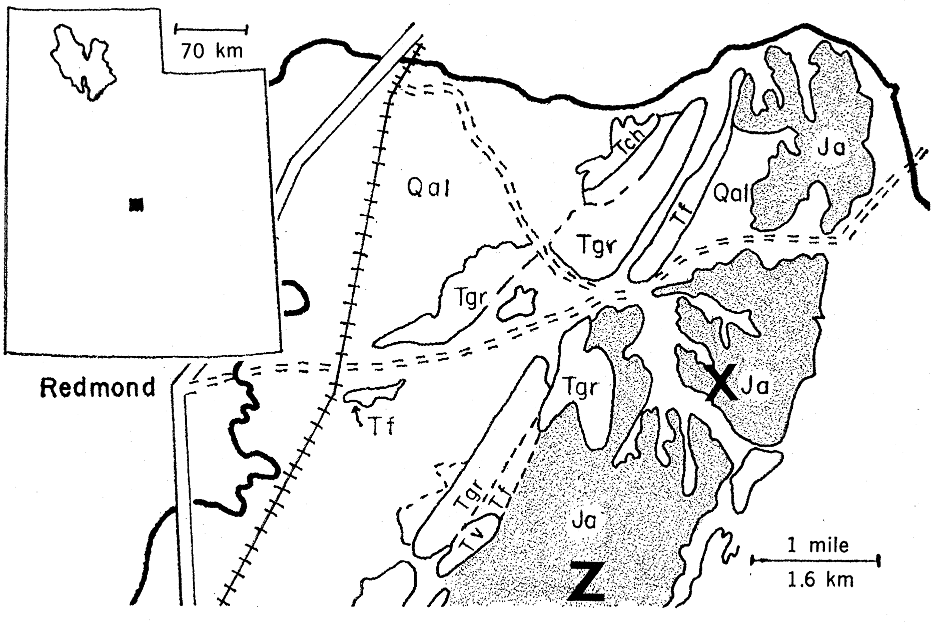

Fig. 1. Index and geologic map showing sites from which the petrified logs of *Palmoxylon pristina* (*X*) and *P. simperi* (*Z*) were collected. *Qal* = Quaternary sediments; *Tv* = Tertiary volcanics; *Tch* = Tertiary Crazy Hollow Formation; *Tgr* = Tertiary Green River Formation; *Tf* = Tertiary Flagstaff Formation; *Ja* = Jurassic Arapien Shale (restricted) [modified after Hardy (*13*)].

only 5 percent of the Lower Cretaceous vascular flora was angiospermous, yet by the end of the Cretaceous, about 95 percent of the world's vascular flora consisted of genera of flowering plants. However, the diversity among even the early Cretaceous angiosperms has caused many to speculate on a pre-Cretaceous origin of the division Anthophyta (Magnoliophyta) (*2, 3*), but there were no unquestionable angiosperm fossils to prove this point.

Axelrod (*3*) reviewed the literature of the supposed Triassic and Jurassic angiosperms in 1952, but Scott *et al.* (*1*) later refuted his examples, and stated rather pointedly (p. 285): "It is probably no overstatement to say that there has not yet been described a pre-Cretaceous plant whose angiospermous affinities are unequivocal." Even as recently as 1968, Dorf (*4*) and Delevoryas (*5*) stated that no pre-Cretaceous angiosperms have yet withstood critical examination. Nevertheless, on strictly theoretical grounds, many authors accept the concept of pre-Cretaceous angiosperms. Cronquist (*6*), for example, writes, ". . . the diversity among these early angiosperm megafossils suggests that the group originated well before Cretaceous time." This is also Axelrod's (*3*) basic conclusion; the origin of the angiosperms, regardless of the known fossil record, is to be sought in the uplands of the Permian.

There are many examples of supposed angiosperms from rocks of Triassic and Jurassic age. The most notable of these is *Sanmiguelia*, a palmlike plant from Triassic deposits in southwestern Colorado (*7*). This fossil consists of leaf impressions with no preserved structure, and many workers feel that such a plant, if it came from rocks of Cretaceous or younger age, would be generally accepted as a palm. Another pre-Cretaceous palmlike fossil is *Propalmophyllum* described by Lignier (*8*) from the early Jurassic of France. However, the fossils are poorly preserved and, to our knowledge, the original specimens have not been critically examined in recent years. One of the oldest reported dicotyledons, *Furcula*, was described (*9*) from the Triassic of Greenland. These specimens have leaf cuticles that are typically dicotyledonous, and they tend to resemble the modern genus *Sterculia*. Regardless, all of these fossils have been seriously questioned as actually being true angiosperms (*1*).

Reports of supposed pre-Cretaceous fossilized angiospermous stems, branch material, or both, are rare. Two genera, *Sahnioxylon* (*Homoxylon*) and *Suevioxylon*, have been considered, but both have failed to meet all the criteria and are now seriously questioned (*1, 10*). Consequently, our discovery of petrified axes in Utah that can be referred to the family Palmae on the basis of their well-preserved cellular structure, is indeed an important find, especially when it comes from Jurassic strata.

Three criteria must be met to substantiate the validity of a proposed pre-Cretaceous angiosperm. First, it must be shown that the fossil was collected in place. Second, the collection site must be demonstrated to be Jurassic or earlier in age. Third, the fossil must be proven to be definitely referable to the Anthophyta (Magnoliophyta), in this case, Palmae.

The petrified palm axes described in this report were uncovered by H. Behunin from the Middle Jurassic Arapien Shale (*11*), approximately 3 miles (5 km) east of Redmond, Sevier County, Utah (Figs. 1 and 2, A and B). Near Redmond, the Arapien Shale

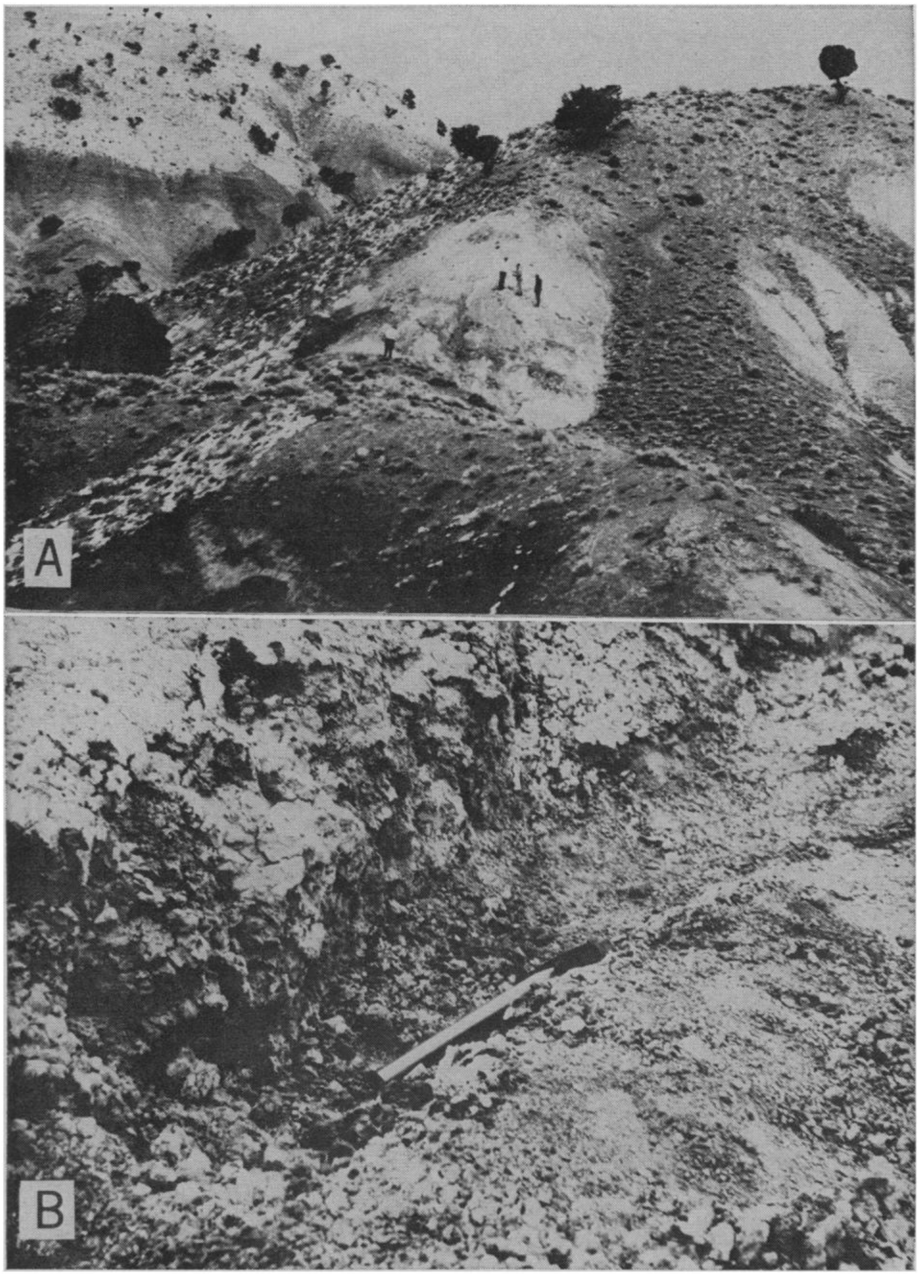

Fig. 2. Collection site of *Palmoxylon pristina* east of Redmond, Utah. (A) Overview illustrating the relationship of the collecting site to the Arapien Shale which forms all of the low rolling hills in the foreground. The actual site is indicated by the figures. (B) Close-up of excavation following removal of the log of *Palmoxylon pristina*.

crops out as low rolling hills which form a part of the western edge of the Wasatch Plateau.

The Arapien Shale was defined (*12, 13*) as a Jurassic formation which appears stratigraphically conformable below the Twist Gulch Formation which, in turn, is overlain in the Salina Canyon region south of Redmond by sediments (*12*) belonging to the Upper Jurassic Morrison Formation (Fig. 3). The Arapien Shale, as described by Hardy (*13*), consists of variegated siltstone, shale, sandstone, halite-bearing beds, limestone, and gypsiferous shale. It is correlated with the Twin Creek Limestone in the Wasatch Mountains and the Carmel Formation of the San Rafael Swell Group. The latter formation interfingers with the Arapien (*11*), and all are of Middle Jurassic age (*14*).

The fossil invertebrate fauna from the Arapien Shale as identified by Reeside (*12*) and Imlay (*13*) and considered by them to be either Upper or Middle Jurassic age, is similar to the invertebrate fauna reported from the lower portion of the Carmel Formation (*13*). During our investigation we found some of the fossil invertebrates previously described (*12, 13*) from the Arapien Shale associated with *Palmoxylon simperi.*

The salt-bearing horizons in the Arapien Shale have been commercially mined east of Redmond. It is from the shales, related to these salt mines, that the specimens of petrified palm axes were collected (Fig. 2, A and B). The specimens consist of two petrified palm logs, one of each species, collected in place, and two additional axes of one species (*Palmoxylon pristina*) found on the surface near the site. Smaller fragments were also obtained from various localities within the formation. Undisturbed gypsiferous shales occur in place immediately above and below the collection site of ***P. pristina*** indicating that the fossil palm axis was in place (*11*).

Palmoxylon simperi was excavated from a contact between undisturbed beds of red and gray shale. In the region of collection, the Arapien Shale is the only exposed formation and fragments of palm axes are continually weathering out of the shales. Younger formations immediately adjacent to the Arapien Shale have been searched to eliminate the possibility that the palm axes may have rolled down from these formations. No evidence of petrified palm specimens was discovered above the Arapien Shale Formation. If one uses Hardy's (*13*) subdivisions of this formation, the collection site of *P. pristina* appears to be in his Unit B which consists of red gypsiferous shale and local salt-bearing horizons.

Palmoxylon simperi Tidwell sp. nov. (Fig. 4)

The petrified axis, approximately 20 cm in diameter by approximately 30 cm long, exhibits an atactostele with randomly oriented bundles. In transverse section individual bundles are essentially ovate with overhanging reniform bundle caps composed of fibers encasing the phloem; bundle sizes range from 1.2 to 1.5 mm in their widest diameter including the bundle cap; 0.74 to 0.93 mm in their narrowest diameter across the conductive portion of the bundle; and the bundle cap width ranges from 1.02 to 1.2 mm. The distance between adjacent vascular bundles varies from 0.09 to 1.1 mm. Circular fibrous bundles, 0.06 to 0.19 mm in diameter, are scattered irregularly among the vascular bundles. The ratio of the number of fibrous bundles to vascular bundles is 2.5 to 1. The ground tissue consists of compact, thin-walled parenchyma cells that are radially expanded with small, narrow, longitudinal lacunae ensheathed among the parenchyma cells. There are from one to four (commonly two or three) metaxylem vessel elements. The largest of these elements is 0.20 mm in diameter; and the smallest, 0.13 mm. In longitudinal section the length of the elements varies from 0.74 to 1.4 mm. Elements have scalariform thickenings on their side walls. Perforation plates are simple on transverse or somewhat oblique end walls. Protoxylem conductive elements vary from 0.037 to 0.10 mm in diameter, and metaphloem elements vary from 0.028 to 0.046 mm. The fibro-vascular (f/v) ratio of the area of the bundle cap to the area of the conductive tissue varies from 1–1.5 to 1. A parenchymatous sheath surrounds the conductive portion of the bundle.

This species was named for our good friend and colleague, A. Daniel Simper, Jr., who has contributed much to this study.

Repository: Brigham Young University, BYU 1378 (holotype).

Palmoxylon pristina Tidwell sp. nov. (Figs. 5 and 6)

The fossil material consists of petrified axes with the largest being about 22 cm in diameter by approximately 1 m long. The axes are atactostelic with bundles randomly oriented. The majority of the bundles are oval in transverse section al-

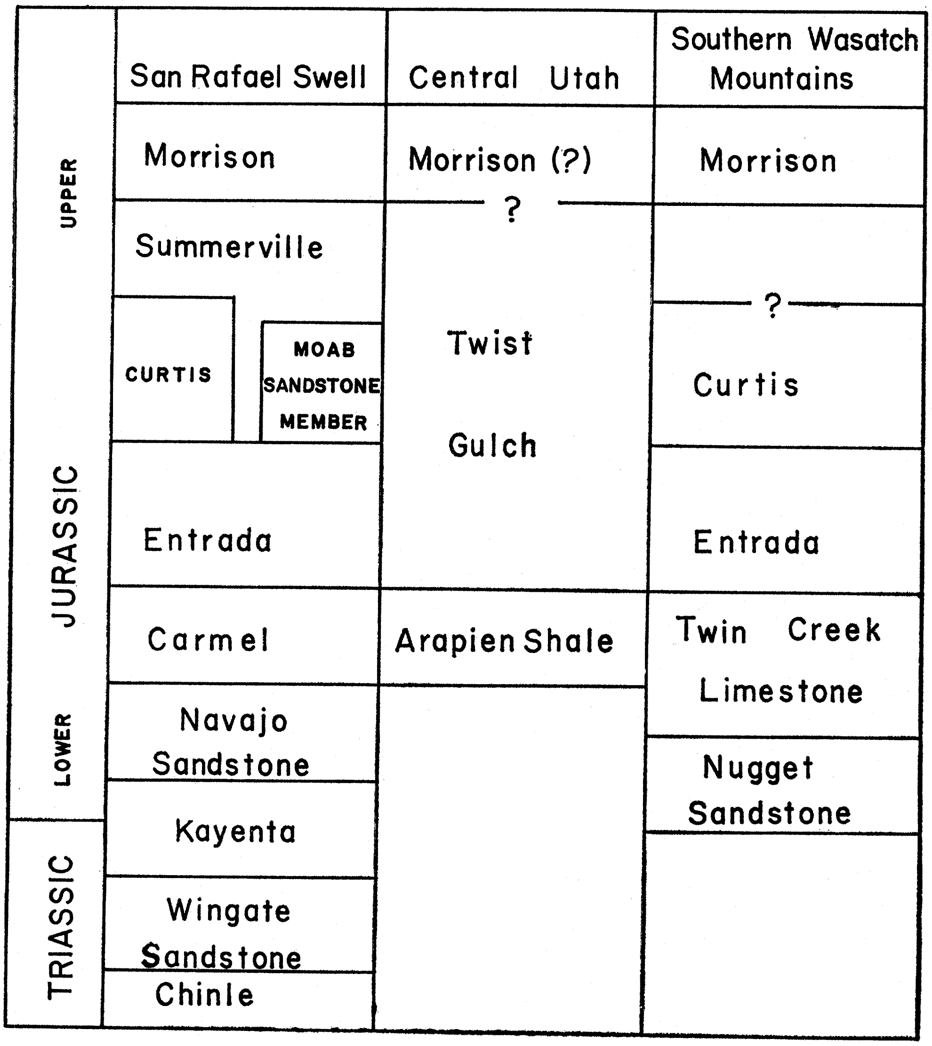

Fig. 3. Stratigraphic chart showing relative correlations between Middle Mesozoic formations in central Utah [modified after Hardy (*13*)].

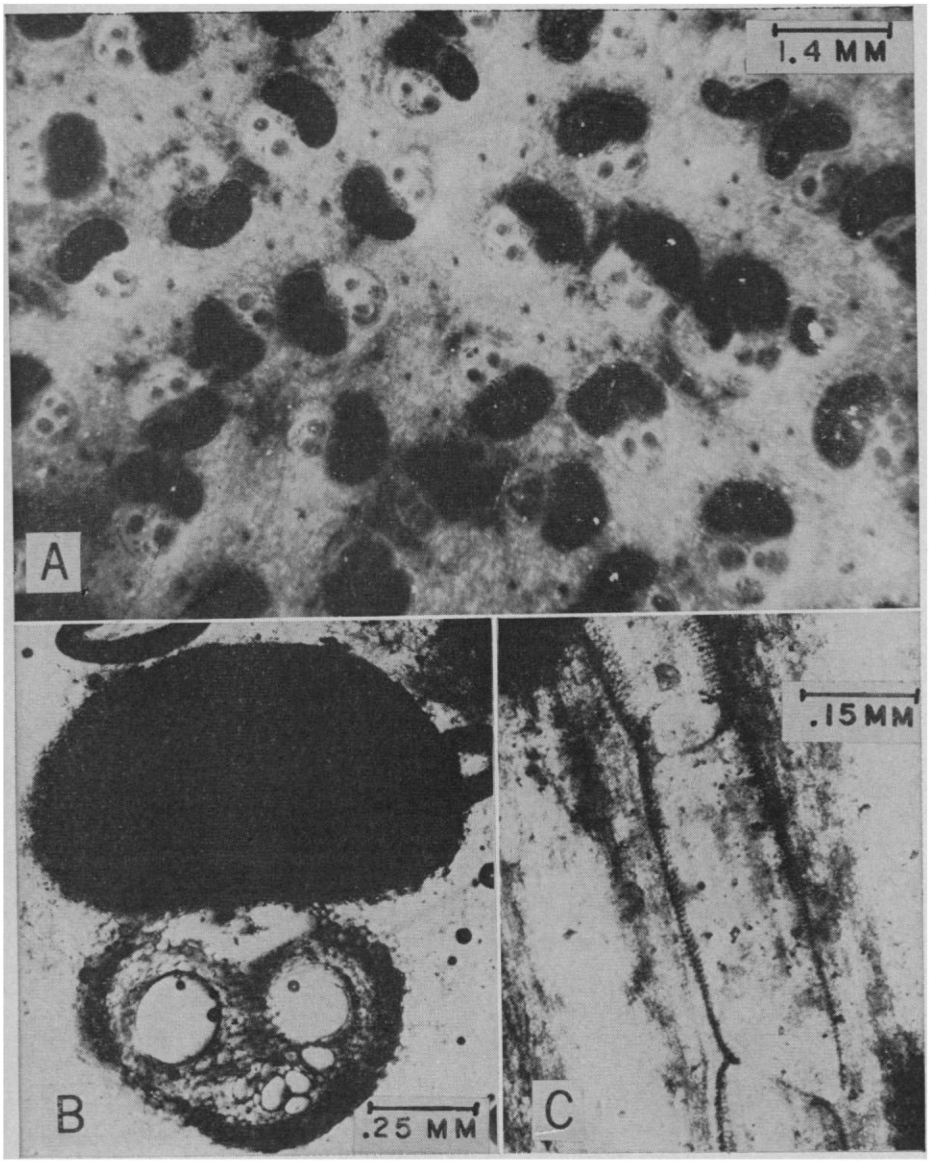

Fig. 4. Vascular bundles of *Palmoxylon simperi.* (A) Scattered bundles. (B) Transverse section of a single bundle of *Palmoxylon simperi* (note the smaller protoxylem elements). (C) Longitudinal section of a vessel showing one vessel element. Note the simple perforation plate.

though some are round to subtriangular. Their sizes range from 0.60 to 0.80 mm in the widest diameter by 0.45 to 0.68 mm in the narrowest diameter. The distance between adjacent vascular bundles varies from 0.10 to 0.65 mm. Circular bundles of fibers 0.04 to 0.59 mm in diameter, are irregularly scattered among the vascular bundles. Ground tissue appears to be composed of compact, thin-walled, nonexpanded parenchyma cells. Metaxylem vessels number from two to five per bundle (commonly two or three). The widest of these vessels is 0.12 mm in diameter and the narrowest, 0.04 mm. Phloem is generally not preserved, although some phloem elements 0.01 to 0.02 mm in diameter have been observed. A reniform bundle cap composed of fibers, 0.20 to 0.38 mm high, is adjacent to the phloem. The f/v ratio in each bundle is 1.7–2.7 to 1. A parenchymatous sheath encloses the conductive portion of the bundle. The ratio of fibrous bundles to vascular bundles is 2.5–3 to 1.

Repository: Brigham Young University, BYU 1377 (holotype).

Palmoxylon simperi and *Palmoxylon pristina* are distinct from one another based upon the shape and size of their respective vascular bundles in cross section and the size of their components. The vascular components are narrower in *P. pristina* than in *P. simperi.* The f/v ratio of *P. simperi* is larger than that of *P. pristina.* The fibrous bundles are generally larger in diameter and more numerous in *P. pristina* than in *P. simperi.*

There is a possibility that *Palmoxylon pristina* and *P. simperi* may represent the same biological species and are from different parts of the plant, but since specimens representing these two species were found widely separated, both geographically and stratigraphically, they are retained as individual species.

In ascertaining and confirming these specimens as being related to Palmae, thin sections of *Palmoxylon simperi* and *P. pristina* were compared with thin sections and with illustrations and descriptions (*15*) of known *Palmoxylon* species of Tertiary age, and generically they are the same. *Palmoxylon simperi* and *P. pristina* are also anatomically similar to living palms, although these similarities are not sufficient for place-

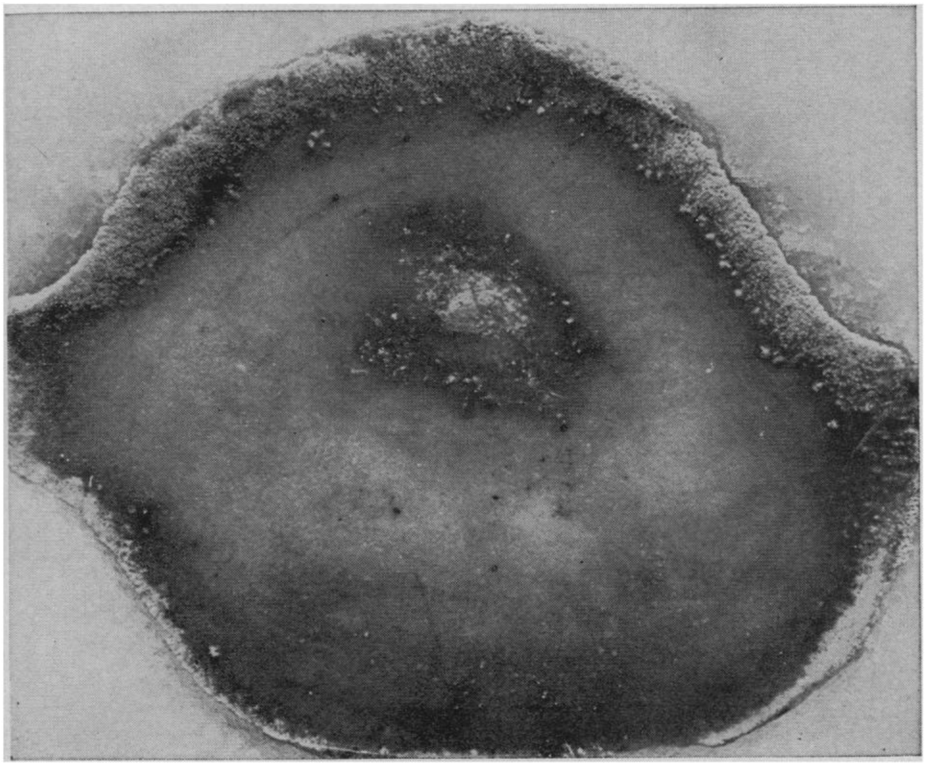

Fig. 5. Cross section of *Palmoxylon pristina* illustrating scattered vascular bundles (× ½).

ment within any extant genus (*16*).

The size of the specimens (up to 22 cm in diameter) of *P. pristina* indicates that they may possibly be stems, although the fiber caps on the vascular bundles are rather narrow for stem material; they are more typical of caps found on the bundles of petioles of the living palms. Metcalfe (*17*) mentions that it is significant that the strands of thick-walled cells scattered among the vascular bundles are composed wholly of fibers. Tomlinson (*18*) finds this characteristic only in *Bactris, Cocos, Erythea, Leopoldinia,* and *Wallichia,* all New World genera except *Cocos* and *Wallichia* (*19*).

Anatomically, petioles of cycads and medullosan seed ferns appear to be similar to the stems of palms, and some investigators have drawn comparisons between them and monocotyledonous stems on the basis of superficial similarities (*20*). *Myeloxylon*, a petiole related to *Medullosa*, has a layer of partially coalesced sclerenchyma strands beneath the epidermis which is lacking in *Palmoxylon pristina* and *P. simperi. Palmoxylon* also differs in bundle composition and lacks the secretory canals present in *Myeloxylon*. The bundles of *Myeloxylon* have a compact mass of tracheids ranging from small to large, and an accompanying strand of phloem separated from the xylem by a thin parenchyma layer. There appears to be no definite fibrous bundle cap in *Myeloxylon* as there is in the palm bundle, but, rather, the caplike structure is composed of phloem—as in the petiole of cycads.

One major difference between cycad petioles and *Palmoxylon pristina* and *P. simperi* is their respective sizes. A *Cycas* petiole is approximately 1 cm in diameter as contrasted to 22 cm for *P. pristina*. The vascular bundles of cycad petioles have a bundle cap composed of phloem. Within these petioles the bundles have a definite arrangement. For example, the bundle arrangement in *Cycas* is somewhat U-shaped, while in *Bowenia* they are arranged as two concentric rings. Xylem elements of bundles in the cycad petiole consist of angular, pitted tracheids, tapering so that in the longitudinal section the tapering ends can be observed. The vessels in both *P. pristina* and *P. simperi*, however, are round and in longitudinal section appear as continuous tubes (Fig. 4C). In fact, the preservation permits viewing the vessel elements and fibrous strands in three dimensions.

The palms belong to a single family, Palmae (Arecaeae). According to Corner (*19*), the extant family is composed of nine subfamilies with some 230 genera containing about 2600 species. On the whole, the family is highly diversified and variable, and the successful placement of fragments such as those found fossilized near Redmond into a genus, is exceedingly difficult even when one applies the criteria of Tomlinson (*18*). Paleobotanists have, therefore, resorted to the employment of organ genera. *Palmoxylon* is applied to the stem and petiole, while *Rhizopalmoxylon* is applied to roots. Leaves and fruits are assigned to several genera with *Palmacites* and *Palmocarpon* frequently being used. In the past, these organ genera have been utilized for fossils from the Cretaceous and Tertiary periods and, because it is impossible to place our material into any modern or other fossil stem genus, we have used the organ genus *Palmoxylon* which now extends back into the Jurassic.

These Middle Jurassic palms, *Palmoxylon pristina* and *P. simperi,* lend some credence to Corner's (*19*) concept

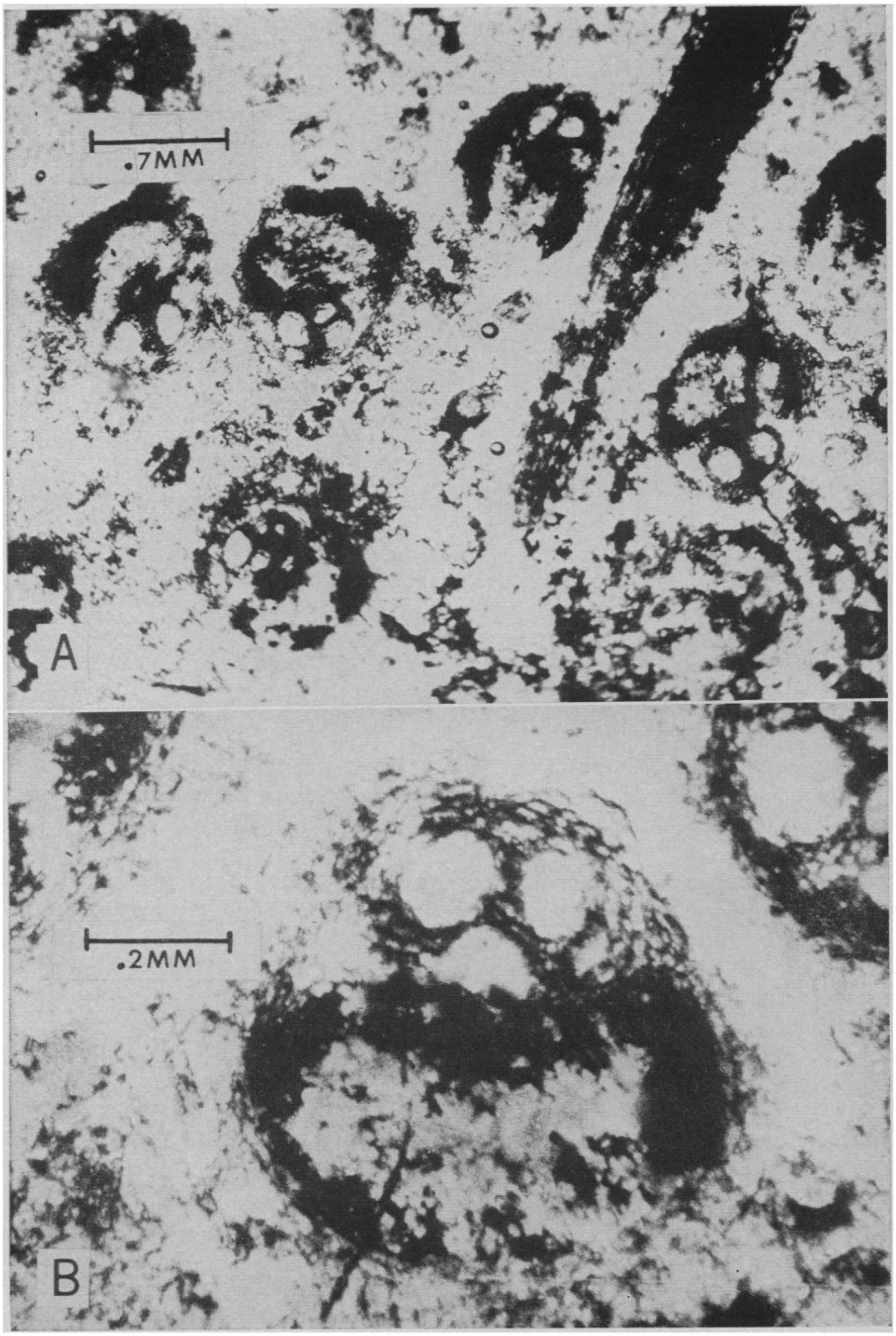

Fig. 6. Vascular bundles of *Palmoxylon pristina*. (A) View of several scattered bundles (note the bundle caps and the vessel elements). (B) Close-up of one bundle.

that the other monocotyledons were derived from palms. He discusses the idea that palms exemplify the "features of monocotyledony" better than any other plants and concludes that while geologically there is little evidence concerning the antiquity of the Palmae, in morphological structure such evidence is found. The palm leaf is thought by Corner to betray in its morphological development the manner in which the monocotyledonous leaf is evolutionarily connected with the dicotyledonous leaf. The palm inflorescence, according to Corner, bespeaks an ancestry greater than the age of known fossil palms. The distribution pattern of the Palmae also suggests the antiquity of this family (*19*).

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16. Slides were sent or shown to several botanists and paleobotanists, many of whom are acquainted with palms, and all were in agreement that these two species are referable to the Palmae. Photographs and the description of *Palmoxylon pristina* and slides of *P. simperi* were sent to C. R. Metcalfe for his evaluation; he and his colleagues agreed that this material is referable to the Palmae.
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21. We thank Dr. C. R. Metcalfe of the Royal Botanical Gardens, Kew, England, for his comments on material sent to him, and Dr. J. Keith Rigby of the Department of Geology, Brigham Young University, for assistance in reviewing the stratigraphy in the field.

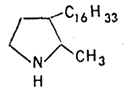
12 January 1970

Alkaloid from Fire Ant Venom: Identification and Synthesis

Abstract. *An alkaloid,* trans-*2-methyl-6-*n*-undecylpiperidine (solenopsin A), has been isolated from the venom of the fire ant* Solenopsis saevissima. *The structure has been confirmed by an unambiguous synthesis.*

Reactions of human beings to the sting of the imported fire ant *Solenopsis saevissima* have demonstrated that the venom of this species is an unusual poison, which, in addition to being a potent necrotoxin (*1*), possesses pronounced hemolytic (*2*), phytotoxic (*3*), insecticidal, and antibiotic activities (*4*). The red form of this ant, which is distributed in the United States from North Carolina to Texas, produces a nonproteinaceous venom which is soluble in organic solvents but insoluble in water (*4*).

In 1966, Adrouny (*5*) proposed structures for the principal toxic components of the venom of the red form of the imported fire ant. The structures proposed were 2-methyl-3-hexadecylpyrrolidine (**1**) and the corresponding Δ^3-pyrroline (**2**). In 1967, Sonnet (*6*) published a synthesis of **1**; gas chromatographic comparison with the ant venom, however, established that neither the *cis* nor the *trans* isomer of **1** was present.



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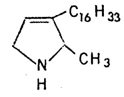

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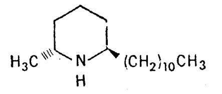
Stimulated by Sonnet's findings, we have undertaken a thorough reexamination of the chemistry of the venom of this ant. We now present evidence that the component of lowest molecular weight (for which we propose the name solenopsin A) is *trans*-2-methyl-6-*n*-undecylpiperidine (**3**, or its mirror image).



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Pure venom was obtained in milligram amounts by "milking" worker ants (*4*); it was stored at about 5°C in hexane solution over anhydrous sodium sulfate. Spot tests suggested the presence of a secondary amine (*7*). The nuclear magnetic resonance (NMR) spectrum (*8*) of the total venom in CCl_4 was τ (tetramethylsilane): 4.73 (distorted triplet, olefinic protons); 6.4 (broad); 6.9 to 7.3, about 8.0, 8.43, and 8.69 (methylene protons); 8.92, 8.98, and 9.08 (triplet, methyl protons). No acidic, aldehydic, aromatic, or *N*-methyl protons were found. Gas chromatographic examination of the total venom indicated the presence of five components with retention times, under the conditions used (*8*), of 6.4, 11.6, 13.0, 23.9, and 27.0 minutes. The mass spectrum of solenopsin A, obtained from combined gas chromatography–mass spectrometry (*8*), exhibited a peak corresponding to the molecular ion at m/e (ratio of mass to charge) of 253 [confirmed by chemical ionization mass spectrometry (CH_4), whose most intense signal was at m/e 254] and signals at m/e 238 (parent ion minus CH_3), 224 (parent ion $- C_2H_5$), and 210 (parent ion $- C_3H_7$), representing cleavage at different points on the methylene chain down to the base peak at m/e 98 ($C_6H_{12}N$—confirmed by high resolution).

Both the mass spectrum and the NMR data indicate the presence of a long alkyl side chain. The carbon skeleton of solenopsin A was determined by the method developed by Beroza (*9*). Only normal hydrocarbons were detected among the hydrogenolysis products from the venom. *n*-Heptadecane was among the products and, for solenopsin A, rules out branching in the alkyl side chain and all substitution patterns on a five- or six-membered *N*-containing ring except for a 2,5-disubstituted pyrrolidine or a 2,6-disubstituted piperidine. The former possibility would require either a 2-ethyl- or a 1,2-dimethylpyrrolidine. The infrared spectrum (no solvent between AgCl plates) (*8*) between 3.35 and 3.42 μm favors