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THE DRISKILL SITE

A Late Woodland Occupation in the Lower Cumberland River Valley

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Introduction

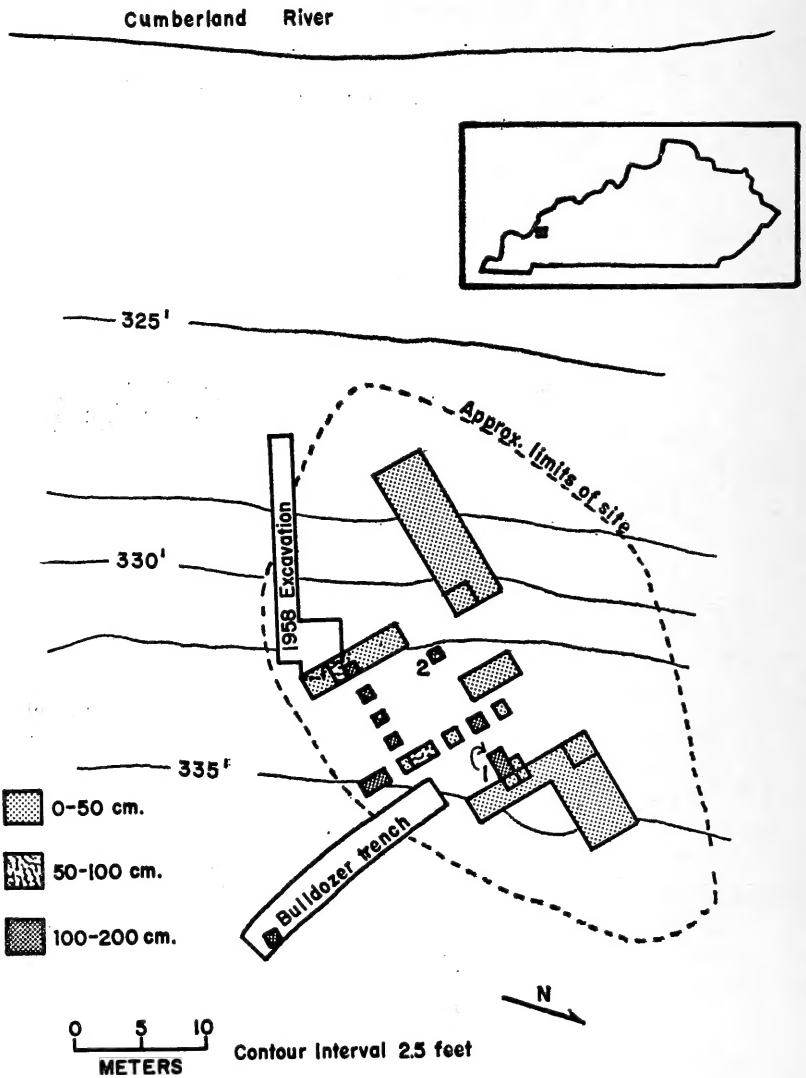
An archaeological survey of the lower Barkley Basin on the Cumberland River in southwestern Kentucky during the winter and spring of 1958 revealed the presence of a Woodland occupation in addition to sites of the Archaic and Mississippian periods (Schwartz, Sloan, and Griffin, 1958). Inasmuch as the Woodland period was virtually unknown in this part of Kentucky, plans were made to excavate the Driskill site (Ly 9) which appeared from the surface to be the largest of the type. In the fall of 1958 preliminary testing revealed clay tempered pottery almost to the exclusion of other ceramic wares. The projectile points on the other hand were all of the small triangular type. Since projectile points of this form are usually associated with the Mississippian period and clay tempered pottery in this area is associated with the Woodland period, it was felt that further work at the site might provide new insights into the relationship between the Woodland and Mississippian complexes. In July of 1959 further excavation was carried on and this paper is the report of that work.

The original survey and all excavation carried out at the Driskill site (Fig. 1) resulted from a cooperative agreement between the National Park Service and the University of Kentucky Museum of Anthropology. This work is part of a larger program of river basin archaeological salvage being carried out by these organizations in an attempt to preserve from destruction some of the archaeological record being destroyed by the dams built for flood control and power purposes.

Geographical Background

The lower Cumberland River valley lies on the western edge of the Mississippian Plateaus characterized by undulating topography with a limestone mantle. The flood plain of the Cumberland River, which in the area of the Driskill site is approximately one mile wide and 100 feet below the surrounding uplands (Fig. 2), is deeply covered by Pleistocene and recent sands, gravels, and silts. Two or three even and regular terraces are usually present paralleling the river. The second of these, on which the Driskill site is situated, may be seen in Fig. 2.

The Cumberland River is the dominant geographic feature of the area. Near the Driskill site it is 400 feet wide and flows at approxi-



mately 27,350 cubic feet per second with a great deal of seasonal variation. This rate of flow was undoubtedly somewhat different in the pre-dam and lock period. Additional water sources are reported in the area in the form of springs in the hills above the river valley.

Average temperatures at the nearest recording station—Paducah—range from 36° F. in winter to 80.8° F. in summer. The effect of summer heat is increased by the high humidity. A great variation in rainfall has been recorded over the past 75 years, ranging from a

minimum of 27.69 inches to a maximum of 72 inches. Average rainfall is 45.35 inches per year.

The Driskill site (Ly 9) lies approximately 100 feet east of the east bank of the Cumberland River some 700 feet south and upstream of the U.S. 62 highway bridge and is situated 6/10 of a mile downstream from Barkley Dam in an area which has now been used as a borrow pit for the dam's earth fill. The site is roughly oval in shape and approximately 495 feet long and 135 feet wide. It stretches from the second river terrace down to the first, and surface indications suggest that the long axis of the site is northeast-southwest.

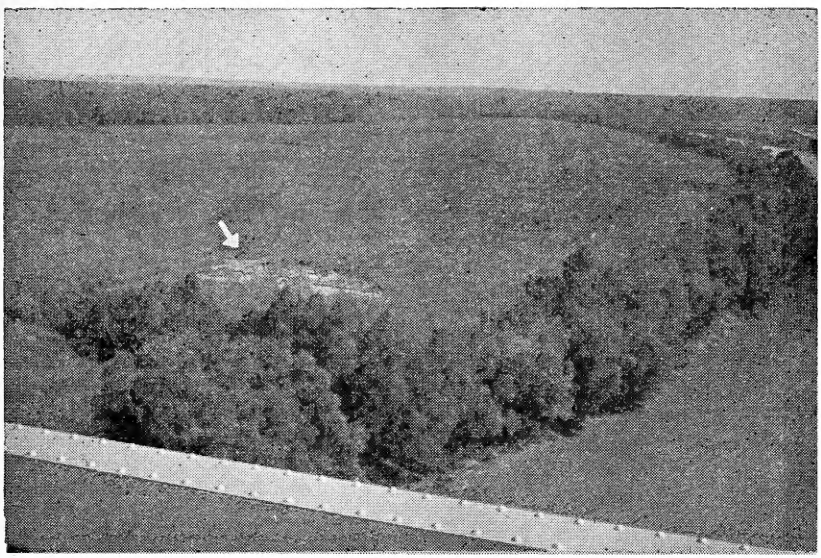


Fig. 2.— Driskill Site and view of surrounding terrain.

Excavation Procedure

The 1959 excavation at the Driskill site began on July 3 and continued through August 8. After preliminary clearing and staking a trench 4 meters wide was begun, coinciding with the long axis of the site, from the top of the second terrace down to the first (Figs. 1, 2 and 3). Excavation was carried on initially by two two-man teams, one man using the shovel while the other screened the dirt. The majority of the squares did not go below 50 centimeters, but several test pits reached 125 centimeters and two were taken to a depth of 200 centimeters (Fig. 1). These pits, some of which were enlarged, were spread out over the site in an attempt to locate a house pattern

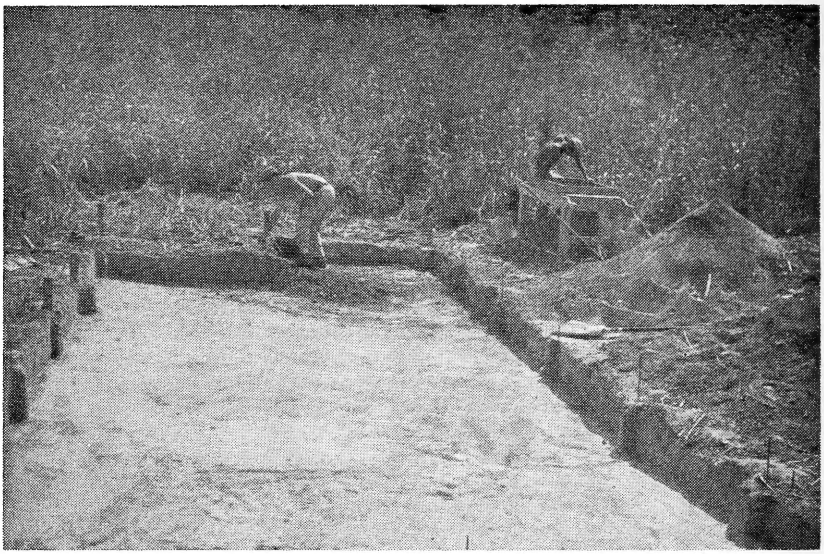


Fig. 3.— Lower trench just before completion and screen in operation.

or other features. As will be noted in the report below, this was an unsuccessful search.

On the final day of excavation a bulldozer was employed to explore a deep section of the upper part of the site over a wide area. A trench 17 meters long and 2 meters wide was cut to a depth of 200 centimeters, but no additional information was gathered as a result of this work.

Results of the Excavation

Two features were recorded from the Driskill site—one possible post hole and an ash lens, but neither are of major consequence. Brief descriptions of these are given below. Their locations can be seen in Fig. 1.

(1) Possible post hole (8L3). The hole extended from 35 cm. below the surface to a depth of 67 cm. It was approximately 8 cm. What appeared to be bark fragments, carbon blackened, were still visibly lining the hole.

(2) Ash lens (18L4). This feature contained two solidified masses of ash, and two limestone rocks, one of which contained hematite. This lens, at 60 cm. depth, was 20 cm. long by 16 cm. wide.

Artifacts

Artifacts found in the course of the excavation were of three general types: chipped stone (84 pieces), ground stone (6 pieces), and pottery (1740 sherds). The bulk of the stone pieces were small, triangular projectile points. Most of the

ground stone items were not distinctive, but the pottery could be easily classified into two main types, Mulberry Creek Cord-marked and Baytown Plain. All of this material is described in detail below.

Stone Artifacts

Projectile Points. Of the 84 pieces of typeable flint found at the Driskill site 76 (90.5%) were projectile points. Seventy-three of these projectile points (96.1%) were triangular, one (1.3%) was corner-notched, and 2 (2.6%) were so broken as to be unidentifiable.

Of the 76 projectile points found (Fig. 4-1), 72 fall into the small triangular classification. This constitutes 85.7% of the typeable flint. Many were broken but enough of a tip of base remained that typing was a simple matter. Ten points were complete enough to measure for length and yielded the following results: range 21.0 to 37.0 mm., with an average of 29.4 mm. Width measurements were possible on 41 points and the range was from 12.0 to 23.0 mm., with the average 17.0 mm. of the 67 points that were complete enough to check, 49 (73.1%) had straight sides, 14 (20.9%) had convex sides, and 4 (6.0%) had concave sides. Forty-seven bases were studied, revealing 27 (57.4%) with concave bases and 20 (42.6%) with straight bases. Seven points (9.7% of the small triangular points) had slight serrations on the sides.

One triangular point was large enough to place in the medium category. Crudely worked into a plano-convex form, this point measured 42.0 mm. in length and 20.0 mm. in width.

Two projectile point fragments (2.4%) were not identified other than that one was a mid-section and the second a tip. Both are from medium-sized projectile points.

Drills. Five of the chipped stone artifacts found were drills, representing 5.9% of the total typed stone artifacts. All of the drills came from the first level. Only two of the drills were complete enough to yield measurements and adequate descriptive information. Both of these are "tear-drop" shaped, the larger measures 52.0 mm. by 19.0 mm. (Fig. 4-2), and the smaller measures 15.0 mm. in width. Two of the drill fragments are tips and one is a mid-section.

Other Chipped Stone. A single blade fragment (1.27%) was found, so broken that neither length nor width could be determined. This fragment appears to be the base of a semi-rectangular blade.

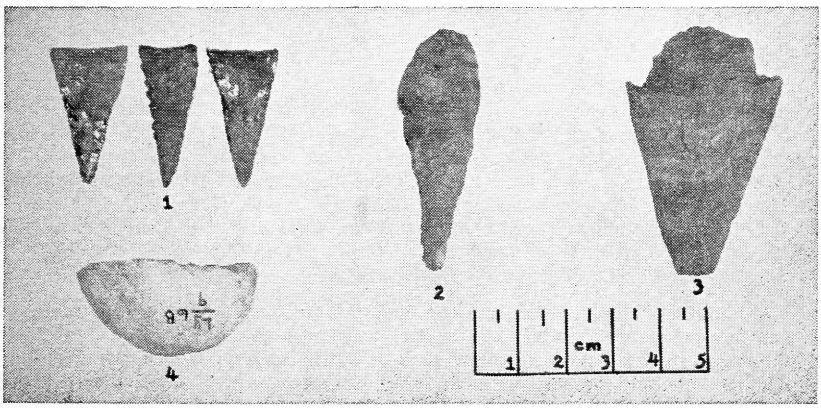


Fig. 4.— Stone sample: (1) small triangular projectile points; (2) drill; (3) corner-notched projectile points; (4) quartz disc.

One crudely worked fragment of the tip end of a scraper was found. No length measurement can be made; the width is 24.0 mm.

A small, irregularly shaped graver (1.27%) was found. This small implement measures 12.0 mm. in width; the base is too broken for accurate measuring.

Twenty-three miscellaneous chipped stone pieces constituted 20.8% of all chipped stone found. Included are fragments from broken artifacts and worked flakes.

Ground Stone. Six fragments of ground stone were found during the excavation. Four were of sandstone and may be from hammer-stones or grinding stones. The only possible measurements are on thickness, which varies from 52.8 mm. to 31.4 mm. Surprisingly, one irregular fragment is banded chert, very sandy in appearance. This piece may have been polished by a stream rather than through use. The sixth piece of ground stone is veined quartz and is from a polished disc. The diameter of the fragment is approximately 39.5 mm. (Fig. 4-4). The edge shown marks of battering such as those found on a hammer-stone.

Table 1.— Stone Summary and Depth Distribution

	Surface 0-25	25-50	50-75	75-100	100-125	Total Number	Percentage
Small triangular points	64	4	....	4	....	72	80.0%
Medium triangular points	1	....	....	....	....	1	1.1%
Corner-notched points	....	....	1	....	....	1	1.1%
Untyped projectile points	2	....	....	....	....	2	2.2%
Drills	5	....	....	....	....	5	5.6%
Blade	1	....	....	....	....	1	1.1%
Scraper	1	....	....	....	....	1	1.1%
Graver	1	1	....	....	....	1	1.1%
Ground Stone	3	1	....	1	1	6	6.7%
	77	6	1	5	1	90	

Discussion of Stone Artifacts

The homogeneity of the stone material, with 80% small triangular projectile points, is unexpected. Only the few drills found make up another category of types that can even be counted as significant. The table of depth distribution needs some explanation, however. Most of the material shown as occurring in the 0-25 cm. level came from the lower slope on the first terrace whereas the material shown below it came from the various deep test pits on the upper slope of the first terrace or the top of the second terrace. Not enough material was found below 25 cm. to draw any conclusions about changes in type frequency, but it should be noted that the main type in the 75-100 cm. level is still the small triangular projectile point.

Pottery

Although 1740 pieces of ceramic material are considered in the following report, several hundred additional sherds were not cataloged because of their minute size. In the section to follow each of the pottery types found is described in detail. Although these types have for the most part been described in print, the types have never been described as they appear in Kentucky. Therefore, more detail is given than might ordinarily have been the case. Two unknown types are briefly described as are the miscellaneous sherds.

Baytown Plain (Phillips, Ford & Griffin, 1951: 76-82) (Fig. 5) 20.2% (350 sherds).

Method of manufacture—coiled.

Temper—predominantly clay; minor amount of sand often as inclusion in paste, shell particles sometimes found.

Texture—sherds tend to chip rather than crumble, and reveal a jagged irregular break.

Color—surface color is predominantly warm gray to buff. Core color differences may not be noticeable, but may be darker than surface.

Thickness—average thickness is 7.0 mm. with a range of 4.0 mm. to 15.3 mm.

Surface finish—most sherds have a chalky feel and are well smoothed on both sides, although the smoothing is present on one surface only (Fig. 10-1).

Rims—Baytown rims often have rim fold (Fig. 5-1) and at least 67% have some kind of decoration, usually notching (Fig. 5-2). Only one rims was rolled outward.

Remarks: The Baytown Plain sherds from the Driskill site display a few differences from the published descriptions, primarily in thickness and percentage of decorated rims.

Two sherds need fuller descriptions: a roughly triangular effigy lug and a node. The lug appears to be the tail of a bird effigy vessel, 42.3 mm. wide, 21.4 mm. long, and 11.9 mm. maximum thickness. Six incised lines appear around the

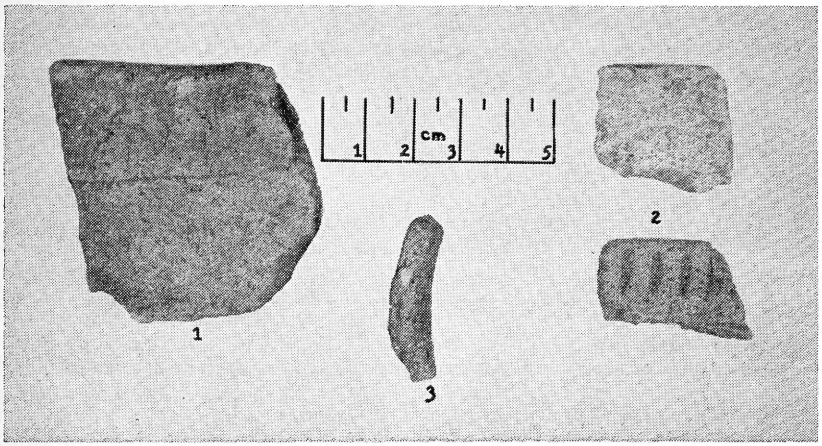


Fig. 5.— Baytown Plain: (1) Rim fold; (2) Plain rim; and notched rim; (3) typical rim profile (outline).

edge opposite the break possibly representing feathers. The lug was apparently added to the rim rather than being a part of the rim coil. The node is a mound on a broader base with four small notches on the upper side of the base. There were probably five notches but a part of the base has been partially destroyed. The node is 16.5 mm. in width and approximately 12.0 mm. in height.

Mulberry Creek Cord-marked (Phillips, Ford & Griffin, 1951: 82-87) (Fig 6) 77.6% (1348 sherds).

Paste—same as Baytown Plain except in thickness; average, 6.4 mm., with a range from 3.6 mm. to 14.8 mm.

Surface finish—cord-marking is present from lip to base, evidently applied by paddle (Fig. 11-1,3,6,7). This marking does not appear to be a decorative technique. Decoration is found on the rim or lip. Many sherds have been so smoothed

after marking that the cord marks are almost obliterated. Cord marks range in size from .4 mm. to 2.1 mm., averaging 1.1 mm. Spacing ranges from 3 to 8 cords per centimeter, averaging 4.8 per centimeter. Cord marks may be vertical (Fig. 11-1, 3) or they may be applied in more than one direction (Fig. 11-6, 7).

Rims—cord-marking is usually found on the rim and often on the lip (Fig.

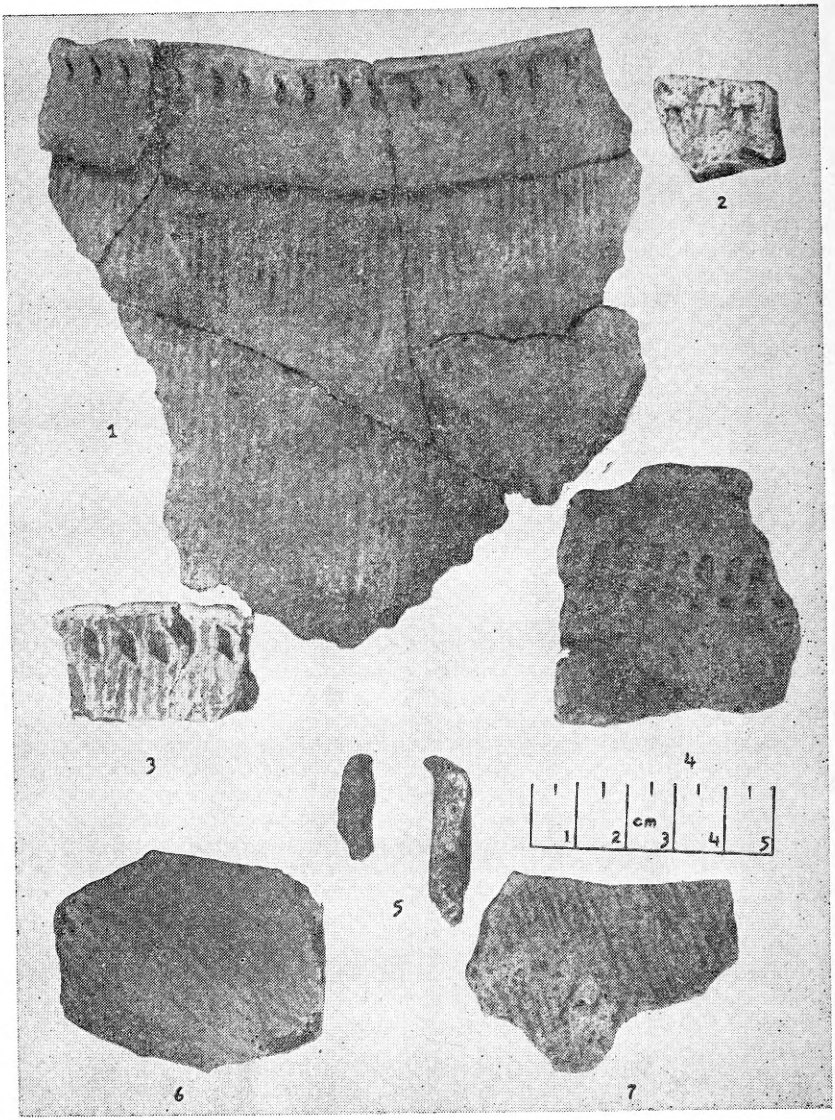


Fig. 6.— Mulberry Creek Cord-marked: (1) cord-marking, rim fold, and punctates; (2) small rim fold or strip, notched; (3) cord-marked, diagonally punctated rim fold; (4) unusual punctuation; (5) typical rim profiles; (6) typical cord-marking; (7) typical cord-marking.

6-1, 3). The rim was usually folded after paddling; the fold was nearly always paddled (Fig. 6-1). Some rims flare slightly outward (Fig. 6-5). Decoration takes the form of nicking, notching, or punctation (Fig. 6-1, 2, 3, 4).

Remarks: These sherds are different in some respects than the published description. Thickness and spacing of cords are the primary variants.

Two sherds will be further described: One appears to be worked along one edge, leaving a marked curve; however, the curved portion is not smooth. A second sherd contains an example of an applied node which was subsequently pinched; "this applique type of pinching, however, is very rare." (Phillips et. al., 1951 p. 86.)

Blue Lake Cord-marked (Phillips, Ford & Griffin: 142-144) 0.7% (12 sherds).

Method of manufacture—coiled.

Temper—predominantly sand; inclusions of clay, clay coarse and lumpy.

Texture—sherds tend to crumble, granular paste.

Color—reddish-gray to dark gray; cores mostly dark.

Thickness—2.0 mm. to 5.6 mm., averaging 4.4 mm.

Surface finish—cord-marking apparently applied by cord wrapped implement (Fig. 13-3). Surface may be rough. Cord size ranged from 1.0mm. to 1.5 mm., averaging 1.2 mm.; cord spacing ranged from 3 to 5 cords per centimeter, averaging 4.2 cords per centimeter.

Remarks: The sherds of this type from Ly 9 are generally dark but fall within the color range of the published description. The main differences appear to be in thickness, size of cords, and cord spacing. No rim sherds were found at this site.

Yankeetown Incised (Blasingham, 1952: 42-44) 0.5% (9 sherds).

Method of manufacture—coiled.

Temper—clay temper; sand occasionally present as inclusion. Clay particles average less than one millimeter in diameter.

Texture—uneven fracture, tend to chip rather than crumble (Fig. 14-2).

Color—surface color is light tan through buff to gray, lighter on the interior surface. Lighter sherds have a matching core; however, the core color of the darker sherd tends to be grayish.

Thickness—range from 4.4 mm. to 6.5 mm., averaging 5.6 mm.

Surface finish—smooth exterior, smoothed or scraped interior.

Rims—one rim sherd was found (Fig. 133-1). On this sherd the decoration described below begins 8 mm. below the lip and continues downward. Three nicks were placed on the upper rim, running slightly onto the lip.

Decoration—consists of parallel incised lines containing diagonal incisions (Fig. 13-1). Although the parallel lines tend to be neatly cut, the diagonal incisions are often rather sloppy—short, long, line not straight, etc.

Remarks: Similar to Baytown Plain paste, and plain sherds may be impossible to separate.

Untyped sherds 1% (17 sherds). Fifteen of these sherds are similar to Mulberry Creek Cord-marked except are somewhat thicker. The cord is only half as thick and the cord marking are further apart. In addition, these sherds appeared slightly darker and contained more imprints in the paste, especially hematite and sand. Two quartz tempered sherds also were dissimilar from other known types.

Shell temper, trace (1sherd). One sherd was probably Neeley's Ferry Plain (Phillips, Ford and Griffin, 1951: 105). Its small size precludes a more definite identification.

Miscellaneous sherds. One sherd is seemingly from the bowl of a ceramic pipe. It measures 15.8 mm. in height; the section of bowl rim is 21 mm. around the exterior rim, 17 mm. around the interior rim, the rim rolls outward, and the interior part of the lip contains 10 parallel nicks. The thickness varies so that a projected bowl diameter may be inaccurate; however, a rough projection yields an

Table 2.— Pottery Summary

Mulberry Creek Cord-marked	1348	77.6%
Baytown Plain	350	20.2%
Blue Lake Cord-marked	12	0.7%
Yankeetown Incised	9	0.5%
Cord-marked, type unknown (clay temper)	15	0.9%
Plain, type unknown (quartz temper)	2	0.1%
Neeley's Ferry Plain (shell temper)	1	T
	1737	
Miscellaneous sherds (1 each: pipe bowl, lug, crazed surface	3	
Total	1740	

Table 3.— Depth distribution only represents sherds in the 200 cm. test pits

8L4	50-75	75-100	100-125	150-175	175-200
Mulberry Creek Cord-marked	5 (62.5%)	11 (68.7%)			
Baytown Plain	2 (25.0%)	5 (31.3%)		1 (25.0%)	
Blue Lake Cord-marked				3 (75.0%)	
Neeley's Ferry Plain	1 (12.5%)				
12L8					
Mulberry Creek Cord-marked		21 (84.0%)	9 (69.2%)	4 (28.6%)	
Baytown Plain		4 (16.0%)	4 (30.8%)	8 (57.1%)	5 (50.0%)
Blue Lake Cord-marked					5 (50.0%)
Untyped-Quartz temper				2 (14.3%)	
Totals					
Mulberry Creek Cord-marked	5 (62.5%)	32 (78.0%)	9 (69.2%)	4 (22.2%)	
Baytown Plain	2 (25.0%)	9 (22.0%)	4 (30.8%)	9 (50.0%)	5 (50.0%)
Blue Lake Cord-marked				3 (16.7%)	5 (50.0%)
Untyped-quartz temper				2 (11.1%)	
Neeley's Ferry Plain	1 (12.5%)				

interior diameter of about 15 mm. and an exterior diameter of about 25 mm. The paste is clay tempered and resembles Baytown Plain.

A single sherd was found which had a crazed surface. It is dark gray to black throughout the paste, yet the exterior surface appears to be a lighter (dirty) gray. The tiny cracks in the surface are quite evident. The temper seems to be a mixture of sand, clay, and quartz.

Most of a lug is attached to a portion of a coil; this sherd was evidently scraped to smooth it. Temper is sand, clay, and quartz, but different from the temper of the crazed sherd. The lug is 16.7 mm. long, 10.9 mm. thick, and an estimated 22.0 mm. wide. The soil is 8 mm. in diameter at the end of the lug.

Discussion of Pottery

As was the case with the stone artifacts, most of the pottery came from the top 25 cm. of the lower slope. In the two deep pits which were put down on the upper slope little material was found until the 75 cm. level was reached (Table 3). In one pit (12L8) no material was found in the top 75 cm. and there was a barren level between 100 cm. and 150 cm. There is a possibility that the barren zones on the top of the second terrace may have resulted in the precipitation of silt after a flood as the water was slowly subsiding. On the other hand when the water subsided from the slope between the second and first terrace the silt would not have precipitated out due to the fast movement of the water.

With regard to the depth frequency of sherds it should be noted that Baytown Plain occurs in the deepest levels with Blue Lake Cord-marked, while Mulberry Creek Cord-marked does not come in until the next level. Also, only in the earliest levels where they occur together does Baytown constitute a higher percentage than Mulberry Creek Cord-marked. It should also be noted that while the single shell-tempered sherd was found in the 50-75 cm. level on the second terrace, this is the first level that sherds were found in this section.

Other Midden Material

Little additional midden material was found at this site. Some two and a half dozen fragments of bone were found, and one piece of a tooth. With the exception of what may be a fragment of a human clavicle, the tiny bone fragments which were burned appear not to be human. The broken tooth seems to be deer or elk. Although most of the bone came from the first level of the lower slope, bits of bone were found in the second, fourth, fifth, and eighth levels.

Tiny, powdery bits of charcoal were found over much of the site. Only a few levels produced charcoal in a size which could be recovered; samples were collected in the first, second, third, sixth, and eighth levels, and from the bulldozer trench.

Three pieces of cannel coal were found in the first level of the lower slope. One piece has a curved edge which could be the result of man's work, but it is too broken to yield further information.

Several charred objects were found in the upper part of the site. In the second level were lumps of burned clay, a piece of burned sandstone, and a fragment of charred nut hull. From the third level came a bit of charred wood, and from the bulldozer trench came burned clay.

Modern artifacts were of two kinds: ceramics from the late 19th or early 20th century, and metal. Sherds of modern ceramics were found in the first level of almost every square excavated. These sherds were all small and were blue-on-white, green-on-white, or blue-and-green-on-white. The metal artifacts consist of seven pieces of iron from the first level of four squares. These bits of metal are scattered at random over the site. Only one tentative identification has been made of a metal object—a musket or rifle ball.

Conclusions

Only two classes of material may used to help determine the temporal range of the Driskill site: chipped stone and pottery. Chipped stone at this site is very homogeneous, with almost all recognizable projectile points being small triangular. This type is usually thought of as being associated with the late pre-historic Mississippian cultures, or the late Woodland cultures. The relative purity of this small tri-

angular type suggests that if this site does fit into the Woodland class, it would be in the later part of it.

Information from the pottery concerning the date of occupancy of this site can be of little more help than the chipped stone. Except for one specimen, none of the sherds were tempered with shell. Therefore an assignment of the site to the Mississippian pattern and time range can be ruled out. The question is then: what light can be shed on the placement of the site from its non-shell tempered pottery?

The high percentage of Mulberry Creek Cord-marked (60% to 70%) over Baytown Plain (25% to 30%) in the upper levels is more or less consistent with the frequency of these types on the northeast side of the Mississippi Valley, as reported by Phillips, Ford, and Griffin (1951:87). Unfortunately, their seriation charts do not go far enough north to overlap the area of this site. Therefore, these major types are of little real value in determining age. It becomes necessary then to search for clues from the lesser types.

The two types which may be useful in dating are Yankeetown Incised and Blue Lake Cord-marked. Yankeetown Incised has been relatively dated from southern Indiana as late Woodland and just pre-Mississippian with perhaps some overlap (Blasingham, 1952). The presence of this type only in the upper levels of the midden in the same position as the one shell tempered sherd implies a similar date for this site, that is, just at the beginning of the Mississippian period. The Blue Lake Cord-marked sherds present an entirely different picture. They consistently occur in the lowest levels of the midden, in layers separated from the upper occupance of the site by sterile alluvial deposits. The time range given by Phillips, Ford, and Griffin (1951:144) for this provisional type is early Baytown, which, according to them, would date about 500 A.D. It should also be noted that in the two deep test pits the levels which had most of the Blue Lake Cord-marked sherds had an entirely different percentage frequency of of Baytown Plain and Mulberry Creek Cord-marked, with the Baytown sherds being at least twice as numerous as the Mulberry Creek. This is almost the opposite of the situation revealed in the upper levels. This, too, would suggest an earlier date for the lower levels of the site.

In summary it can be said that the Driskill site was probably occupied at least twice during the Baytown or Woodland period, the earliest use probably about 700 A.D. This date is based on the presence of Blue Lake Cord-marked, the higher percentage of Baytown Plain, the lack of shell tempered pottery, the lack of Yankeetown Incised, but the presence of small triangular projectile points. Without the latter the date would have been somewhat earlier. The second period of occupation is dated at about 900 A.D. on the basis of the presence of Yankeetown Incised, Neeley's Ferry Plain (one sherd), the

lack of earlier types as found in the lower levels, and the great number of small triangular projectile points.

The length of the hiatus between these two occupations is not known, but its presence is evident from the twenty-five centimeters of sterile alluvium between them. The change in frequency of the major pottery types does imply a significant period of elapsed time, however, as does the presence of different minor pottery types.

The Driskill site can be affiliated culturally with the broad Woodland pattern of eastern United States prehistory. This assignment is based on the type of pottery, its temporal position, and the lack of certain large structural features such as temple mounds which would infer a Mississippian placement. The temporal range evident from the artifact occurrences suggests that Woodland occupancy of the lower Cumberland River Valley lasted over a relatively long period of time. The fact that no burial mounds or other features similar to the Adena culture of central Kentucky were found in the valley during the survey implies that the Woodland occupation of Kentucky can be divided into at least two major aspects. Both of these may have developed from an Archaic base, but they apparently followed distinctive paths—the Adena developing a materially flamboyant culture, while its contemporaneous Woodland cousins to the south simply added pottery and a few other traits to their Archaic base and finally made what may have been a rocky transition to Mississippian culture.

Until further excavation in western Kentucky Woodland sites no more inferences should be made. Additional information is badly needed on house patterns, a wider range of ceramic and stone artifacts, and burial customs. With this data further comparisons, with phases such as Lewis and Dillinger in southern Illinois, can be made. There is a definite similarity between all these late Woodland manifestations, but their interconnections can only stand as problems until further work is done.

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THE APPLICATION OF THE MANNICH REACTION TO KOJIC ACID

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The Mannich reaction involves a condensation of ammonia or an amine with formaldehyde and a compound having at least one reactive hydrogen atom. Among the latter are such compounds as aliphatic and aromatic ketones, substituted beta keto acids and esters, aliphatic aldehydes, acetylene compounds, and phenols. One of the major qualifications which a compound must possess in order to undergo the Mannich reaction is that of containing a hydrogen of pronounced activity. Recently (1), it has been shown that kojic acid possesses such activity with one of its hydrogen atoms.

A classical example of this activity of hydrogen involves the condensation of a carbonyl compound, such as acetonphenone, with formaldehyde and an amine, according to the following reaction:

$$C_6H_5COCH_3^{*} + HCHO + R_2NH \cdot HCl \rightarrow C_6H_5COCH_2CH_2NR_2 \cdot HCl + H_2O$$

(Active hydrogens are starred: *)

Perhaps the earliest record of a so-called Mannich-type condensation may be found in the German dye literature. Bayer and Co. (2) in 1897 treated phenols and napthols with amines and formaldehyde. They believed, however, that the products formed were ether derivatives rather than *alpha*-amino-cresol derivatives.

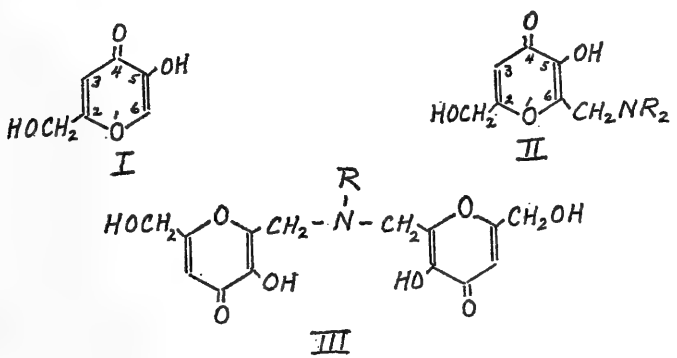
Tollens and van Marle (3) in 1903 were able to isolate a product from the reaction cited in the reaction above which proved to be a tertiary amine, but they failed to recognize the reaction as a general one. Later, this reaction was extended by Auwers and Dombrowski (4) who demonstrated that dialkylaminomethylphenols could be obtained from a condensation utilizing formaldehyde, secondary amines and phenolic compounds. The reaction then captured the attention of Mannich (5) who began a systematic and detailed study of the reaction which now bears his name.

Kermach and Muir (6), attempting to find new anti-malarials, found that hydrogens in the methyl group of quinaldine hydrochloride

were sufficiently reactive to form definite compounds with HCHO and diethylamine or piperidine. Decombe (7) later prepared Mannich derivatives of several phenols and showed that amino substituents entered the ring *orpho* or *para* to the hydroxyl group with monohydroxy phenols. In 1939, Caldwell and Thompson (8) produced further evidence for the substitution of a Mannich group directly on the phenolic ring by obtaining nuclear methylation from the hydrogenolysis of the Mannich derivative of *sym*-xlenol. Bruson and MacMullen (9) submitted evidence for poly Mannich group substitution by introducing three such groups into phenolic rings using either morpholine or piperidine.

Burckhalter (10) demonstrated the physiological activity of Mannich-substituted phenols in the preparation of some anti-malarial compounds. His work also included chloroquinolines and acridines (11). Burke (12, 13, 14) was able to produce compounds of the benzoxazine type by use of primary amines with phenols.

Interest in producing compounds with varying degrees of physiological activity has resulted in the synthesis by Meadow and coworkers (15, 16, 17) of a large number of amino derivatives of phenols, substituted phenols, and bisphenols by employing some modifications of the Mannich reaction. In 1959, O'Brien, Patterson and Meadow (1) showed that a Mannich-type reaction can be applied to kojic acid, 5-hydroxy-2-hydroxymethyl-4*H*-pyran-4-one, and several aliphatic and heterocyclic amino derivatives of this acid were reported. The results of this work indicated that in kojic acid the active hydrogen for Mannich purposes was in position 6 of the kojic acid ring (I), and Mannich substituents formed compounds corresponding to formulas (II) and (III). In the case of primary aliphatic amines used, both hydrogens attached to the nitrogen were replaced by kojic acid rings as in (III). For secondary amines, derivatives corresponding to the structure (II) were obtained.



The method of Meadow and Reid (15) and also that used by Woods (18) led to the formation of resinous materials when kojic acid was heated with formaldehyde and strongly basic amines. In view of the sensitivity of the pyronone ring to basic reagents (19) it is understandable that the Mannich reaction as applied to kojic acid and basic amines has not been widely studied. Thus kojic acid, although capable of entering into some of the substitution reactions typical of a phenol, is not as stable under these conditions as a true phenol. The success of our study depends on the fact that kojic acid was found to take part in the Mannich reaction under mild conditions which did not produce extensive ring cleavage; in fact, kojic acid reacted rapidly with morpholine and other basic amines in the presence of formaldehyde at room temperature. In all cases, products were isolated which contained a single Mannich group in the 6 position of kojic acid. Attempts to force a second group presumably into the 3 position failed.

Continuation of the work on kojic acid has led to the preparation in this laboratory of four new mono-Mannich derivatives of kojic acid, using mildly basic aromatic amines. These compounds are described in Table I. In the case of the primary amines *p*-chloroaniline, *o*-aminophenol, and *p*-aminophenol, both hydrogen atoms attached to the nitrogen were replaced by kojic acid rings as shown in structure (III) above. With 1,2,3,4-tetrahydroquinoline, however, a derivative whose structure is similar to (II) was obtained.

Discussion of Results

Effect of Varying the Nature of the Amine Component of the Mannich Reaction

The amine components which we have studied show a wide variation in basicity. In general, the reactivity of the amines with kojic acid was found to increase with increase in basicity, so that more strenuous reaction conditions were required for the weakly basic amines. In addition, the sensitivity of kojic acid to ring cleavage was greater for the more basic amines, making milder reaction conditions necessary. For amines of comparable basicity, the reaction conditions were essentially the same.

The heterocyclic and aliphatic amines were strongly basic and appeared to react rapidly, so that it was possible to prepare derivatives at room temperature. Prolonged heating of the reaction mixture caused extensive decomposition with formation of highly colored materials. The conditions normally used for preparing Mannich derivatives of a true phenol (15, 16) could not be applied. In the case of a true phenol, the formation of phenol-formaldehyde condensation products is minimized by using a large excess of amine, with respect to

formaldehyde, and by adding the latter slowly to a cold mixture of the phenol and amine. After standing at room temperature for an hour to permit formation of an amine-formadlehyde intermediate, the mixture can be heated to reflux temperature for several hours to permit maximum conversion to a Mannich derivative.

The decomposition of kojic acid in the presence of amines is a side reaction which must be minimized if the preparation of Mannich derivatives of this acid is to be successful. This was accomplished by adding the kojic acid to a mixture of the amine and formaldehyde in equimolar quantities as nearly as possible.

The weak basicity of the aromatic amines made it possible to heat the reaction mixture for a period up to 20 or 30 minutes without causing serious decomposition of kojic acid. The fact that kojic acid was used in excess with respect to the amine, in order to substitute both hydrogen atoms of the amine group, was an added factor which favored greater stability of the kojic acid ring. The products obtained were sufficiently stable to permit prolonged heating in alcohol during recrystallization.

The use of *o*- and *p*-aminophenol in the Mannich reaction with kojic acid provided good evidence of the contrast in properties which exists between kojic acid and a true phenol. In this reaction, kojic acid behaved as the "active hydrogen" containing component of the Mannich reaction and the aminophenol as the amine. The kojic acid gave a relatively pure product in good yield, despite the fact that it was in competition with the active *ortho* and *para* hydrogen atoms of the aminophenol. It is evident that the reactivity of kojic acid in the Mannich reaction is significantly higher than the reactivity of a true phenol. The substitution of both amine hydrogen atoms of an aminophenol by kojic acid is somewhat surprising, and is indeed indicative of the pronounced activity of kojic acid in this type of reaction.

Experimental

All melting point determinations were made with a Fisher-Johns apparatus, and are uncorrected. The Kjeldahl analyses for nitrogen were determined by a modification of the method of McKenzie and Wallace (20), with 0.025 N sulfamic acid as the titrant.

1. *p-Chloroaniline Mannich Derivative of Kojic Acid.* Two and six-tenths grams (0.02 mole) of *p*-chloroaniline was mixed with 3.6 g. (0.044 mole) of 27% aqueous formaldehyde solution and 40 ml. of 95% ethanol. The reaction mixture was heated for 15 minutes, after which 6.4 g. (0.044 mole) of kojic acid was added. The resulting mixture was then heated for an additional 15 minutes, and allowed to

stand at room temperature for one hour. Water was added to produce a noticeable turbidity, and the mixture was chilled overnight. The solid product was filtered off and recrystallized from 50 ml. of hot 95% ethanol, adding a small amount of water until turbid, and chilling in a refrigerator. The yield after one recrystallization was 5.0 g. (58% of pink needles, melting at 192-193° with decomposition. A neutralization equivalent could not be obtained for this product, but the Kjeldahl nitrogen analysis indicated that both hydrogen atoms of the amine group of *p*-chloroaniline had been substituted by kojic acid rings.

Anal. Calcd. for $C_{20}H_{18}ClNO_8$: N, 3.21. Found: N, 3.12.

2. *Preparation of o-Aminophenol Mannich Derivative of Kojic Acid.* Three and five-tenths grams (0.025 mole) of kojic acid was mixed with 1.1 g. (0.01 mole) of *o*-aminophenol and 25 ml. of 95% ethanol, after which 2.4 g. (0.03 mole) of 37% aqueous formaldehyde was added. The mixture was allowed to stand 30 minutes at room temperature and then boiled for 15 minutes. Water (about 50 ml.) was then added to the reaction mixture to cause turbidity. After chilling overnight, 2.8 g. (67%) of orange colored crystals, m.p. 119-120°, were obtained. The product was recrystallized from 40 ml. of hot 95% ethanol to which some water had been added. One recrystallization produced 1.5 g. of crystals, melting at 120-121°. The analysis indicated that both hydrogen atoms of the amino group in *o*-aminophenol had been replaced by kojic acid rings.

Anal. Calcd. for $C_{20}H_{19}NO_9$: N, 3.36. Found: N, 3.12.

Note: in order to prove that this product was actually a Mannich derivative, a sample of the material was decomposed in the following manner. A 0.2 g. sample of the *o*-aminophenol Mannich derivative was boiled with 10 ml. of conc. NH_4OH for 10 minutes in a 50 ml. Erlenmeyer flash. After adding 30 ml. of water, heating was continued for 45 minutes, taking care not to evaporate the solution to dryness. The dark brown solution was cooled, diluted to 20 ml. with water, and extracted with four 20 ml. portions of ether. The ether was then washed with 10 ml. of water and allowed to evaporate slowly on a watch glass, obtaining a small amount of tan crystals, m.p. 164-170° with decomposition. This material produced a brown color with ferric chloride identical to that given by a sample of *o*-aminophenol. A mixture of the material with authentic *o*-aminophenol melted at 164-170°.

The treatment with ammonia decomposed the kojic acid rings in the Mannich derivative. The fact that *o*-aminophenol could be recovered after this alkaline decomposition indicates that there had been no nuclear substitution of the aminophenol by action of formaldehyde.

3. *p-Aminophenol Mannich Derivative of Kojic Acid.* In a manner very similar to that for the preparation of the derivative from *o*-aminophenol, 3.5 g. (0.025 mole) of kojic acid was treated with 1.1 g. (0.01 mole) of *p*-aminophenol and 2.4 g. (0.03 mole) of 37% aqueos formaldehyde. The product yield was 3.7 g. (88%), melting at 111-113°. The yellow product was recrystallized from hot 50% aqueous ethanol and chilled to produce 2.0 g. of crystals, m.p. 115-116°. Analysis indicated that also in this case both hydrogen atoms in the amine group had been replaced by kojic acid groups.

Anal. Calcd. for $C_{20}H_{19}NO_9$: N, 3.36. Found: N, 3.50.

Note: A 0.2 g. sample of this Mannich derivative was decomposed with ammonium hydroxide by the same procedure used for the degradation of the Mannich derivative of *o*-aminophenol. After evaporation of the ether extract, 0.04 g. of tan crystals, melting at 180-193° with decomposition, was obtained. A mixed melting point with pure *p*-aminophenol was not depressed (181-183°), thus indicating that no nuclear substitution of the *p*-aminophenol had taken place and that active hydrogens in the Mannich condensation had been furnished by kojic acid.

4. *Preparation of 1,2,3,4-Tetrahydroquinoline Derivative of Kojic Acid.* Two and seven-tenths grams (0.02 mole) of 1,2,3,4-tetrahydroquinoline was mixed with 1.6 g. (0.02 mole) of 37% aqueous formaldehyde and 25 ml. of 95% ethanol. The mixture was boiled for 15 minutes, after which 2.8 g. (0.02 mole) of kojic acid was added and the mixture allowed to stand overnight at room temperature. Water was then added to cause turbidity, and the solution was chilled to cause crystallization to take place. A yellow product weighing 2.5 g. (44%), m.p. 134-138°, was obtained. The melting point was raised to 138-139° after recrystallization from hot aqueous ethanol. The product was somewhat unstable and the recovery after recrystallization was less than 50%. Prolonged heating during recrystallization therefore should be avoided.

Anal. Calcd. for $C_{16}H_{17}NO_4$: N, 4.87. Found: N, 4.74.

5. *Reaction of Kojic Acid with Formaldehyde and p-Aminobenzoic Acid.* It was not possible to obtain a Mannich derivative of *p*-aminobenzoic acid. The only indentifiable product collected was a yellow solid, melting at 193-195°. On the basis of nitrogen analysis and its neutralization equivalent, it is believed that this substance is N-(hydroxymethyl)-4-aminobenzoic acid. The neutralization equivalent was determined by titration with sodium hydroxide to a phenolphthalein end point.

Anal. Calcd. for $C_8H_9NO_3$: N, 8.32; neut. equiv., 167. Found: N, 8.53, neut. equiv., 167.

Table 1.— Mannich Derivatives of Kojic Acid With Aromatic Amines

Mannich Derivative	Formula	Melting Point °C	Nitrogen %	
			Calc.	Found
p-Chloroaniline	$C_{20}H_{18}ClNO_8$	192-93[a]	3.21	3.12
o-Aminophenol	$C_{20}H_{19}NO_9$	120-21	3.36	3.12
p-Aminophenol	$C_{20}H_{19}NO_9$	115-16	3.36	3.50
1,2,3,4-tetrahydro-quinoline	$C_{16}H_{17}NO_4$	138-39	4.87	4.74

[a] Melted with decomposition.

Summary

Kojic acid has been shown to possess an active hydrogen in the 6 position which enables it to undergo reactions of the Mannich type in the presence of formaldehyde and amines. Mild conditions were necessary for the success of this reaction. Aliphatic amines caused rupture of the kojic acid ring at elevated temperatures, while a short period of heating was necessary for the weakly basic aromatic amines. Four new Mannich derivatives of kojic acid with aromatic amines are described.

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Lexington, Kentucky.

ANNOTATED CHECKLIST OF FISHES FROM CLEMONS FORK, BREATHITT COUNTY, KENTUCKY

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Introduction

Clemons Fork of Buckhorn Creek lies in eastern Breathitt County within the large tract known as Robinson Forest. The entire area, which is controlled by the University of Kentucky, is relatively undisturbed and almost completely covered by second growth hardwoods. Clemons Fork consists of a trunk stream, about two and a half miles long, and many short tributaries. The basin is too small, approximately 6000 acres, to maintain flow during periods of drought, but the numerous sandstone and gravel-bedded pools offer permanent habitat.

Use of Robinson Forest for field biology courses and projects has stimulated investigations of the vertebrate fauna. Barbour (1956) enumerated the birds along Clemons Fork, and Bush (1959) has reported on the herpetofauna. This article is a further contribution to these efforts.

The checklist is based on fishes seined during the summer of 1959 and the spring of 1961. Most specimens are deposited in the University of Kentucky collections. The arrangement of families, genera and species is essentially that used by Moore in Blair, et al. (1957). Considering its small size, Clemons Fork has a rich fish fauna. Seven widely-scattered collection stations yielded seventeen species. Common names used in this account are those suggested by the American Fisheries Society (1960).

Species Account

FAMILY CATOSTOMIDAE

Catostomus commersoni (Lacépède). A single specimen of the white sucker was taken in the lower end of Clemons Fork. The species achieves moderate abundance downstream.

Hypentelium nigricans (Le Sueur). Eleven hog suckers were caught from larger pools in lower Clemons Fork. This species is the typical sucker of the stream.

FAMILY CYPRINIDAE

Semotilus atromaculatus (Mitchill). The creek chub lives in extreme headwaters to the exclusion of other forms and is common throughout the basin. Hundreds were netted but only 49 were kept.

Notropis ardens (Cope). Six rosefin shiners were taken from pools of the trunk stream. The fish is more common in larger creeks.

Notropis cornutus (Mitchill). The common shiner is abundant in the lower half of the main stream. Twenty-seven individuals were retained, though several times that number were captured.

Notropis spilopterus (Cope). The spotfin shiner is rare in Clemons Fork, though common in larger creeks of the region. Only two specimens were obtained.

Ericymba buccata (Cope). Nineteen specimens of the silverjaw minnow were collected in the lower half of the main stream. The species frequents exposed, shallow pools.

Pimephales notatus (Rafinesque). Seven individuals of the bluntnose minnow were taken in large, silted pools.

Campostoma anomalum (Rafinesque). The stoneroller is not so abundant as the creek chub, nor does it invade extreme headwaters. More than a hundred were caught, but only twenty-nine were retained.

FAMILY CENTRARCHIDAE

Micropterus dolomieui Lacépède. Smallmouth bass are the characteristic top carnivore in streams of the region, but Clemons Fork has only a few pools deep enough to maintain the adult. Single adult specimens were taken at two points in the main stream.

Lepomis megalotis (Rafinesque). In these restricted headwaters the longear sunfish is rare. A single specimen was taken, though the species is common in adjacent stream systems.

Ambloplites rupestris (Rafinesque). The rock bass is typical of swift permanent streams but does occur in Clemons Fork. One adult was taken in a large pool, which also contained a smallmouth bass.

FAMILY PERCIDAE

Etheostoma blennioides Rafinesque. Five greenside darters were taken from larger riffles in the lower reaches of the main stream. This area seems to be the upstream limit of distribution.

Etheostoma caeruleum Storer. Except for extreme headwaters, the rainbow darter can be found in most riffles in Clemons Fork. A dozen specimens were retained and several others were released.

Etheostoma nigrum Rafinesque. The Johnny darter is a common inhabitant of pools throughout the basin. Thirty-four specimens were retained and an equal number released. Below the headwater pools, it is the most common darter in the basin.

Etheostoma sagitta (Jordan and Swain). The arrow darter is typical of headwater pools and decreases in abundance downstream. Since this species has been considered rare, all thirty-four specimens captured were retained. The taxonomic status, habits, and peculiar distribution of the species are discussed by Kuehne and Bailey (1961).

Discussion

The fishes collected from Clemons Fork represent seventeen species from only four families. Specific variety and abundance are surprisingly good, considering the small size of the watershed. The creek chub, stoneroller and arrow darter typify headwater collections. Highly predaceous fishes are restricted to a few, large pools. Some species, such as the white sucker, spotfin shiner, and greenside darter, occur only rarely in lower Clemons Fork but are common downstream.

Good forest cover and lack of disturbance to the watershed contribute to more stable stream conditions than are found in most parts

of eastern Kentucky. The abundance of fishes and the predictability of their occurrence are thought to be correlated with habitat stability.

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