

Identifying white oak logs with sodium nitrite

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Abstract

Because red oak logs can spread oak wilt, the European Economic Community (EEC) requires that all oak logs imported from the eastern United States where oak wilt is found must be fumigated. However, a Common Market Commission has indicated that logs of white oak may be exempt from fumigation if "a reliable method for identifying white oak from other species has been developed for use by officials at the ports of unloading." We found that spraying a 10 percent solution of sodium nitrite on the heartwood of oak logs is a simple color test that unerringly separates the eastern U.S. red oaks from the white oaks. To confirm the accuracy of this chemical identification test, we tested nearly 10,000 oak logs at 30 sawmills throughout the eastern United States. In addition, we tested nearly 500 eastern oak specimens and all commercially important eastern hardwoods and softwoods from the Forest Products Laboratory wood collection. In every test on oak heartwood, the sodium nitrite indicated the correct species group. Of the other species, only chestnut (*Castanea*) and occasionally chinkapin (*Castanopsis*) reacted like white oak. As with nearly all chemical reactions, temperature affects the reaction time and development of diagnostic color.

The aim of this study was to establish a simple, reliable test that officials could use at ports of unloading to distinguish white oak¹ logs from other species, particularly red oak.²

European Economic Community (EEC) regulations require that oak logs imported from areas in the eastern United States where oak wilt (*Ceratocystis fagacearum* (Bretz) Hunt) is found must be fumigated, a costly and potentially hazardous process. Researchers, however, have reported that white oak (unlike red oak) could

rarely, if ever, transmit oak wilt from the United States to Europe (4, 5, 7, 8, 15). In response to these findings, the Commission of the EEC has indicated that logs of white oak may be exempt from fumigation if "a reliable method for identifying white oak from other species has been developed for use by officials at the ports of unloading" (2).

Macro- and microscopic methods of distinguishing between red, white, and live³ oak groups now exist (14, 19) that are accurate and efficient, but they require a high level of expertise and are much too time consuming and troublesome when hundreds or thousands of logs need to be identified quickly.

Stearns (17, 18) developed a quick, easy, and accurate chemical method of separating the red and white oaks using a mixture of benzidine and sodium nitrite (NaNO₂). Stearns and Hartley (18) state that a 10 percent sodium nitrite solution used alone would also distinguish the red oaks from the white oaks, but the distinction was "not quite as sharp as the benzidine-nitrite mixture." The advantage of sodium nitrite alone was the longer shelf life.

¹Ring-porous species of oak (*Quercus*) in the subgenus *Lepidobalanus*, also known as *Leucobalanus*.

²Ring-porous species of oak (*Quercus*) in the subgenus *Erythrobalanus*.

³Diffuse-porous species of oak (*Quercus*) that are in both subgenera *Erythrobalanus* and *Lepidobalanus*.

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Willeitner et al. (20) studied Stearns' methods on European oaks and additionally used sodium nitrite plus o-anisidine (1) and sodium nitrite plus aminobenzosulfonic acid. The concluded that a 10 percent sodium nitrite solution used alone was accurate 95 percent of the time, and the addition of benzidine, o-anisidine, and aminobenzosulfonic acid did not enhance the distinction.

In cooperation with the Federal Research Organization for Forestry and Forest Products, Institute of Wood Biology and Wood Preservation, Hamburg, Federal Republic of Germany, we tested these and other potential chemicals on a limited number of specimens and determined that the best was sodium nitrite. We then extensively tested sodium nitrite on samples in wood collections and at various sawmills throughout the eastern United States and on the docks at Norfolk, Virginia, and Bremerhaven, Federal Republic of Germany.

Materials and methods

Chemical selection

We selected chemicals used by Stearns and Hartley (18) and Willeitner et al. (20): o-anisidine-sodium nitrite, benzidine-sodium nitrite, and sodium nitrite. Since these chemicals were originally used as heartwood-sapwood indicators (10), we selected additional heartwood-sapwood indicators from Kutscha and Sachs (11): alizarine-iodine, alizarine red S (0.75%), Benedict's solution, bromcresol green (0.0413%), bromphenol blue (0.0413%), Fehling's solution, ferrous ammonium sulfate (0.1 N) ferrous sulfate (0.1 N), hydrochloric acid-methanol, iodine (2.5%), methyl orange (0.1%), and potassium iodide-iodine. Except for sodium nitrite, these chemicals were only tested on samples cut from eight logs of the "select oaks" (6) (Table 1), called "select" because of their commercial importance based on the volume of wood produced and the quality of the product. To gather testing specimens, foresters in Wisconsin, Mississippi, Indiana, and Iowa collected 3- to 4-foot-long logs no less than 12 inches in diameter from trees of these select oaks. To assure a positive identification, twigs with leaves and/or flowers and fruits were also collected.

Preliminary testing with sodium nitrite

To determine the best testing method, various preliminary tests using sodium nitrite were conducted on specimens cut from the select oak logs that included the following:

1. Tests at moisture conditions from green to 6 percent.
2. Tests at 1, 5, 10, and 20 percent sodium nitrite with tap and distilled water.
3. Tests with various application methods and quantities of solution.
4. Tests on samples varying in size from logs, lumber, and veneer to chips, particles, sawdust, and wood flour.
5. Tests at temperatures from -20°C (-4°F) to 27°C (80°F).

TABLE 1.—List of eastern United States oaks tested with sodium nitrite from the Madison (MADw) and Samuel J. Record (SJRw) wood collections.

Scientific name ^a	Common name ^a	Commercial ranking ^b	No. of specimens
RED OAKS (<i>Erythrobalanus</i>)			
<i>Quercus</i>			
<i>arkansana</i> Sarg.	Arkansas	N	1
<i>coccinea</i> Muenchh.	Scarlet oak	C	17
<i>ellipsoidalis</i> E.J. Hill	Northern pin oak	C	2
<i>falcata</i> Michx.	Southern red oak	C	22
<i>falcata</i>			
var. <i>pagodifolia</i> Ell.	Cherrybark oak	S	8
<i>georgiana</i> M.A. Curtis	Georgia oak	N	2
<i>ilicifolia</i> Wangerh.	Bear oak	N	4
<i>imbricaria</i> Michx.	Shingle oak	C	9
<i>incana</i> Bartr.	Bluejack oak	N	8
<i>laevis</i> Walt.	Turkey oak	N	14
<i>laurifolia</i> Michx.	Laurel oak	C	22
<i>marilandica</i> Muenehh.	Blackjack oak	C	18
<i>myrtifolia</i> Willd.	Myrtle oak	N	4
<i>nigra</i> L.	Water oak	C	21
<i>nuttallii</i> Palmer	Nuttall oak	C	2
<i>palustris</i> Muenehh.	Pin oak	C	10
<i>phellos</i> L.	Willow oak	C	13
<i>rubra</i> L.	Northern red oak	S	40
<i>shumardii</i> Buckl.	Shumard oak	S	18
<i>velutina</i> Lam.	Black oak	C	34
WHITE OAKS (<i>Lepidobalanus</i>)			
<i>Quercus</i>			
<i>alba</i> L.	White oak	S	46
<i>bicolor</i> Willd.	Swamp white oak	S	13
<i>chapmanii</i> Sarg.	Chapman oak	N	2
<i>durandii</i> Buckl.	Durand oak	N	4
<i>lyrata</i> Walt.	Overcup oak	C	20
<i>macrocarpa</i> Michx.	Bur oak	S	20
<i>michauxii</i> Nutt.	Swamp chestnut oak	S	6
<i>muehlenbergii</i> Engelm.	Chinkapin oak	S	8
<i>oglethorpensis</i> Duncan	Oglethorpe oak	N	1
<i>prinus</i> L.	Chestnut oak	C	48
<i>stallata</i> Wangerh.	Post oak	C	30
<i>stallata</i>			
var. <i>margaretta</i> (Ashe) Sarg.	Sand post oak	N	2
<i>stellata</i>			
var. <i>paludosa</i> Sarg.	Delta post oak	C	1
<i>virginiana</i> Mill.	Live oak	C	19
<i>virginiana</i>			
var. <i>geminata</i> (Small)			
sarg.	Sand live oak	N	2

^aAccording to Little (13).

^bS = select oak according to Cooper and Watt (6), C = commercial oak according to Little (12). N = noncommercial oak according to Little (12,13).

Red oak was not tested at various temperatures because there was no way to gauge the slight color changes. Samples from the white oak disks were placed in temperature-controlled rooms, refrigerators, or shaded outdoor areas to obtain a range of temperatures. After applying the 10 percent sodium nitrite solution either by hand sprayer or eyedropper, we periodically observed the samples to determine when the color reaction reached completion. In cold or freezing conditions we also heated the sodium nitrite solution to near boiling (90°C) before application, heated the surface of the disk, and interpreted the results before the reaction reached completion.

Testing specimens in the wood collections

To determine if all species of the eastern U.S. oaks tested correctly, we tested specimens from the Madison (MADw) and Samuel James Record (SJRw) wood collections located at the Forest Products Laboratory. We

tested 491 specimens representing 35 species and varieties, 20 red oaks, 13 white oaks, and 2 live oaks, based on information from Little (12, 13) and Cooper and Watt (6) (Table 1). Specimens without heartwood or those with decay were excluded.

We were curious to determine how toher oaks from throughout the world and other species besides oak (particularly eastern U.S. species) tested. So we selected and tested wood specimens from our wood collections of all the important eastern U.S. hardwoods and softwoods (following Little(12)), some western species, and many oaks from throughout the world.

Field testing oaks

We also tested oaks at sawmills and at ports of loading and unloading. At sawmills we tested nearly 10,000 logs where we only knew whether the logs were red or white oak and not the exact species. In all tests we used a 10 percent solution of sodium nitrite and applied the chemical with a 1-quart (liter) and sprayer. This sprayer had the capacity to test hundreds of logs, and one could test over 100 logs in less than an hour. Logs stacked 10 to 15 feet high were easily reached with the spray. These logs were tested during the summer months (temperatures between 20° to 30°C (68° to 86°F)) at sawmills throughout the eastern United States and on the docks at Norfolk, Virginia, and Bremerhaven, Federal Republic of Germany. A total of 30 mills were visited in Wisconsin, Indiana, Kentucky, Tennessee, Alabama, Mississippi, Georgia, Virginia, Pennsylvania, Maryland, New Hampshire, New York, and Vermont, and near the city of Hamburg, Federal Republic of Germany. During visits we assumed that oak logs of all commercially important species were represented and that they were cut from different habitats and were in various conditions of freshness.

Weathered logs with gray ends and logs that were end coated with wax were difficult to test. Sometimes the color reactions seemed to occur, but as a result of the gray background or wax coating the color change could not be accurately evaluated. Removing a small portion of the weathered wood exposed the normal wood which then reacted typically. Generally this weathered surface was less than ¼ inch deep even on badly weathered logs. An ax, hatchet, chainsaw, rasp, lathe tool, or knife can be used to freshen the surface, but we found that a chinsel works best.

Results and discussion

Preliminary chemical testing

During preliminary chemical testing on the select oaks, sodium nitrite and sodium nitrite in combination with benzidine and o-anisidine clearly distinguished the red and white oaks. Benzidine, however, has a "high" Toxicity Hazard Rating (THR) and is a recognized carcinogen (16). Less is known about o-anisidine, but it is also suspected carcinogen (Dr. Po-Yung Lu, personal communication). Since no differences in color, intensity, reaction time, or other properties were noted between sodium nitrite and sodium nitrite in combination with these carcinogens (confirming the findings of Willeitner et al. (20)), we eliminated testing

with the carcinogenic compounds and only used sodium nitrite for extensive testing. In the crystalline or concentrated form sodium nitrite is a strong oxidizing agent and warnings on the label should be followed. As a 10 percent solution sodium nitrite is relatively safe, but eye and skin contact and inhalation should be avoided by proper safety equipment.

The heartwood-sapwood indicators (11) did not identify red and white oak, but most did delimit the heartwood-sapwood boundary. In particular, alizarine red S (0.75%) and methyl orange (0.1%) were excellent heartwood-sapwood indicators in both red and white oak.

Preliminary testing with sodium nitrite

Color sequence.—Using a 10 percent solution of sodium nitrite (100 g/L or 95 g/qt), we sprayed disks cut from logs of the select red and white oaks. Figure 1 shows the series of color changes on both red and white oak. When the photo was taken, the disks were at room temperature and at near-green moisture condition. On the white oak, the heartwood area sprayed with sodium nitrite immediately turns yellowish or brown with an orange tint. In 1 minute the color begins to darken, and within 3 minutes the color is dark orange brown or reddish brown to oxblood or carmine. Within 5 minutes the color is dark greenish or purplish brown to black. In 2 hours the color has faded and in 16 hours it is brownish. On the red oak, the sprayed heartwood area turns yellowish or brown with an orange tint, but the color does not turn appreciably darker with time. After 2 hours, the color has faded to a greenish yellow and in 16 hours is a light yellowish brown. In several days the color on both red and white oaks has faded so that no distinction between the two can be made. On both the red and white oaks the sapwood and bark are essentially unchanged.

Effects of moisture content.—At moisture conditions from green to 6 percent, only slight changes occurred in reaction time or in the color series. In green or fresh material the reaction time seemed faster than in material dried to 6 percent moisture content. In addition, the orange shades seemed more pronounced in fresh material, whereas in drier material a more yellowish or greenish tint was noticeable. Also, the colors on fresh material seemed sharper and more distinct than on dried material. However, the final color was always the same.

Effects of concentrations.—No differences were detected with tap and distilled water and between the 5, 10, and 20 percent solutions of sodium nitrite. At the 1 percent level, the reaction did occur and a positive test could be ascertained, but the color series was less intense and the reaction time was longer. Although the 5 percent can be used, we recommend the 10 percent solution to ensure sufficient chemical when used on green and wet logs.

Effects of application methods.—Various application methods and quantities of sodium nitrite solution produced the same results. However, depending upon the type and size of specimen and the location of

the test, different methods of application worked better than others. One-liter hand sprayers purchased in hardware stores were excellent for field testing logs or lumber. When spraying the 10 percent solution, avoid eye contact and inhalation by use of chemical safety goggles and mask. Eye droppers or glass rods were best for lab tests or when accuracy of application was necessary.

Effects of specimen size.—Large and small pieces of oak reacted in essentially the same length of time and produced the same color sequence. In wood flour, however, the reaction time was slightly faster, and the final color of the red oak was greenish brown, but it was still quite obvious from the color reaction which samples were white oak and which were red oak.

Effects of temperature.—We tested the select white oak disks in temperatures from -20°C (-4°F) to 27°C (80°F). The appearance of the dark greenish brown or black color indicated that the reaction had reached completion. Several independent tests were run at the same temperatures with different observers since minor color distinctions are sometimes subject to individual interpretation.

At 27°C (80°F) the reaction only takes 5 minutes, but as the temperature is lowered the reaction takes longer. At 10°C (50°F) the rate is 20 minutes, and at 0°C (32°F) the rate is approximately 60 minutes. Below freezing, the rates are much longer, almost 24 hours at -20°C (-4°F). These rates, however, are based on using a cold solution and attaining a completed reaction.

In order to shorten the time necessary to make a positive identification between red and white oaks, especially in cold or freezing weather, we heated the sodium nitrite solution and/or surface of the disk and interpreted the results before the reaction reached completion. If we used a hot (90°C) sodium nitrite solution on cold or even frozen samples, the rate of reaction increased. It also increased if the surface of the sample was heated with a hair dryer, hot iron, or blow torch. If both were heated, the reaction rate greatly increased. Of course, the degree to which the reaction rate increased depended upon the temperature of the heated solution at application, the temperature of the wood surface, and the air temperature. At temperatures near freezing, we could halve the time necessary to bring the reaction to completion.

With a little experience in interpreting the color test results, we did not have to wait until the reaction was complete to make a positive identification of white oak. The reddish oxblood or carmine colors typical of the white oak color pattern (Fig. 1) are seen much earlier in the reaction. At -20°C (-4°F) and colder we noticed that when using a cold solution the reddish color develops in several hours. With a hot solution, the reddish color appears in less than 15 minutes. The completed reaction, however, takes hours since the surface and/or solution is cooled to the air temperature.

Chemical reaction.—The chemical compound(s) in white oak that react with sodium nitrite to produce the color reaction are not known. We suspect, however, that

the reaction is related to a color test originally devised by Procter and Paessler for ellagic acid in Gupta et al. (9). Bate-Smith (3) and Gupta et al. (9) used this test, a spray reagent of nitrous acid, in paper chromatographic analysis to detect esters of hexahydroxydiphenic acid and their derivatives. According to Gupta et al. (9) "Esters (of hexahydroxydiphenic acid) react with nitrous acid to give initially a carmine-red changing through green, brown, and purple to finally (5 to 10 min.) a moderately stable indigo-blue color."

Testing specimens in the wood collections

Specimens in the white and live oak groups from the eastern United States tested positive (i.e., dark greenish brown to black), and those in the red oak group tested negative (i.e., yellowish or orange brown to brown) (Fig. 1). On those rare occasions when labeled red or white oak specimens did not test as expected, we examined the specimen with a hand lens and found that the specimen was mislabeled. Thus, the sodium nitrite test correctly indicated the proper oak group for all the specimens tested from the eastern United States.

Species of oak from throughout the world were also tested. Most white and red oaks reacted as expected, but tests on live oaks (diffuse-porous species) were mixed. Species of live oaks occur in both the subgenera *Lepidobalanus* and *Erythrobalanus*. For example, live oak (*Q. virginiana* Mill.) from the eastern United States and covey oak (*Q. copeyensis* C. H. Muller) and roble negro (*Q. oleoides* Schlecht and Cham.) from Central America are in the subgenus *Lepidobalanus*, whereas roble (*Q. seemannii* Liebmann) from Central America is in the subgenus *Erythrobalanus*. A few specimens of these species which were backed by herbarium material and identified by a reputable taxonomist were tested. Those species in subgenus *Lepidobalanus* reacted like white oak while *Q. seemannii* (*Erythrobalanus*) reacted like red oak. In other cases, where the exact species was not known, some live oak specimens did not react with the sodium nitrite as expected. Because it is impossible to correctly identify the proper subgenus for the live oaks based solely on the wood anatomy, we cannot determine whether or not a doubtful reaction reflects a misidentification or variability. We suspect that most live oaks in *Lepidobalanus* and *Erythrobalanus* react like white and red oak, respectively. Additional testing and careful examination will hopefully clarify the problem.

Of all the species tested other than oak, only species of chestnut (*Castanea*), in the oak family (Fagaceae), always reacted like white oak. Species of chinkapin (*Castanopsis*), which is also in the oak family, occasionally reacted like white oak. However, we are not certain whether the chinkapin specimens that reacted negatively contained heartwood or whether they were mislabeled.

Field testing oaks

All red and white oak logs tested in the field reacted as expected (Fig. 1). Occasionally logs in the white oak stack or deck tested like red oak or vice versa. Upon checking the identification with a hand lens, however, we found that in every case the questioned log was

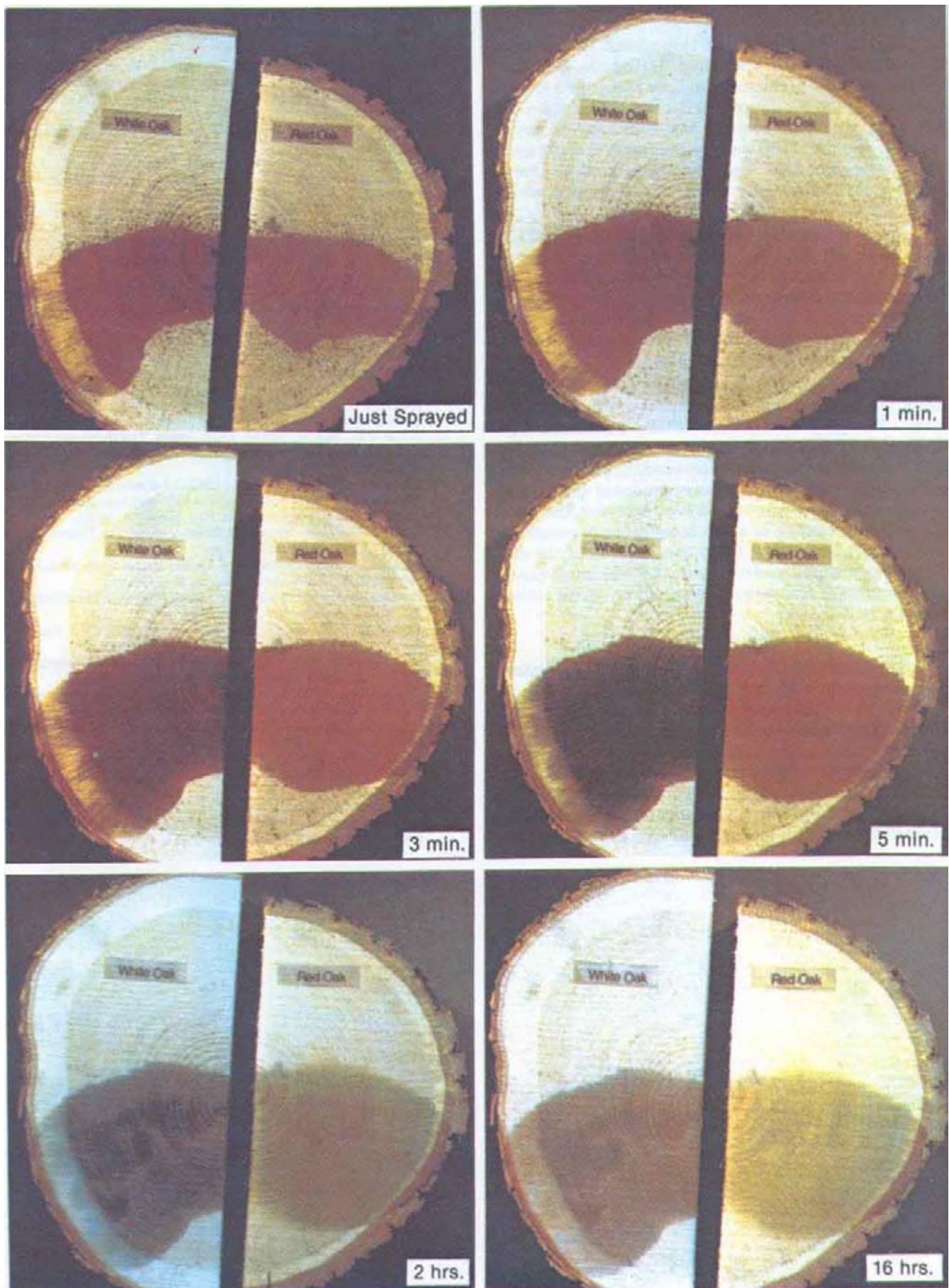


Figure 1.—Color sequence using 10 percent sodium nitrite on green disks of a white oak (swamp chestnut oak—*Quercus michauxii* Nutt.) and a red oak (cherrybark oak—*Quercus falcata* Michx. var. *pagodifolia* Ell.).

placed in the wrong stack. In some cases we even found non-oak species. Thus, the sodium nitrite indicator was never wrong.

Most visits to sawmills throughout the eastern United States were conducted during the summer months. Generally the reaction time to completion was approximately 5 minutes, and never more than 10 minutes. Fresh or recently cut logs reacted more quickly, and the colors were more intense than on end-checked or drier logs. The differences, however, were minimal and essentially could be disregarded while field testing.

Areas in logs with obvious decay or mineral stain sometimes did not react as expected. Normal areas within the same log, however, did give typical color changes. Since decay and mineral-stained areas can be avoided and because logs with these defects are low quality and not exported, their identification should not concern European port officials.

Weathered and wax-coated logs sprayed with sodium nitrite often did not produce typical color reactions. The gray background of the weathered logs masked the true color, and the wax prevented the solution from contact with the heartwood. Exposing a fresh surface and then testing always produced the typical reaction. On some wax-coated logs, however, the coating was thin enough to allow some spray to penetrate and produce a reaction. Of course, because of the distinctive colors produced in white oak it was much easier to determine if the spray penetrated on white rather than on red oak logs.

Conclusions

Sodium nitrite in a 10 percent solution is a chemical which produces a distinctly different color reaction on red and white oak heartwood in about 5 minutes at 27°C (80°F). This chemical test distinguishes white oaks from all important eastern US. hardwoods and softwoods, except chestnut, and is very accurate and reliable. Officials at ports of unloading or other personnel with little or no background in wood identification could easily be trained to identify white oak logs using this chemical.

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