PREPARATION OF X-RAY NEGATIVES OF TREE-RING SPECIMENS FOR DENDROCHRONOLOGICAL ANALYSIS

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ABSTRACT

Techniques for producing X-ray negatives of dendrochronological specimens have been developed at the Geological Survey of Canada and the Nondestructive Testing Laboratory, Mines Branch, The radiographs are produced to provide tree-ring density data to supplement ring-width measurements for dating and climatic studies. New specimen preparation techniques and X-ray methods are discussed. The quality and quantity of tree-ring information is enhanced by the use of X-ray analysis.

INTRODUCTION

The usefulness of tree-ring density analysis for evaluating the commercial quality of wood and for dendroclimatic interpretation has been demonstrated in recent years by Polge (1965a, 1965b, 1966), Green and Worrall (1964), Green (1965), and Harris (1969). One of the best techniques for obtaining graphic and quantitative tree-ring density data is to scan X-ray negatives of dendrochronological specimens on a densitometer (Polge 1966; Jones and Parker 1970). The purpose of this paper is to describe the technique used at the Geological Survey of Canada to prepare X-ray negatives of tree-ring samples for dendrochronological analysis. Particular attention is given to the production of radiographs that can be used for the two traditional foci of dendrochronology: (1) the dating of tree-ring specimens, and (2) the relating of tree-ring data to climatic factors.

Accurate crossdating of the annual rings in tree-ring series and the assigning of correct calendar year dates to each ring in tree-ring chronologies is essential for both dendrochronological dating and dendroclimatic analysis. Tree-ring density measurements obtained from X-ray negatives can be used to supplement ring-width measurements for crossdating purposes (Jones and Parker 1970). Tree-ring chronology characteristics such as false annual rings, locally absent rings, rings with very faint latewood, and microscopic rings are important considerations in crossdating ring widths. These same characteristics need to be considered in tree-ring density studies and X-ray examples of these features are presented.

Techniques in field collection and specimen preparation used at the Geological Survey of Canada are designed to accommodate both tree-ring density and tree-ring width analysis. Preparation requirements are more stringent for density studies and new specimen preparation methods have been devised. Specimen preparation and X-ray exposure procedures are designed to accommodate large quantities of material.

Two techniques for producing the X-ray negatives have been developed. In one method the X-ray film, the tree-ring specimen, and the X-ray source are held stationary during exposure. This technique is similar to that used by Polge (1966) in his tree-ring density studies. In the other method the X-ray source is held stationary and emits radiation through a narrow slit onto a moving carriage supporting the tree-ring specimen and film.

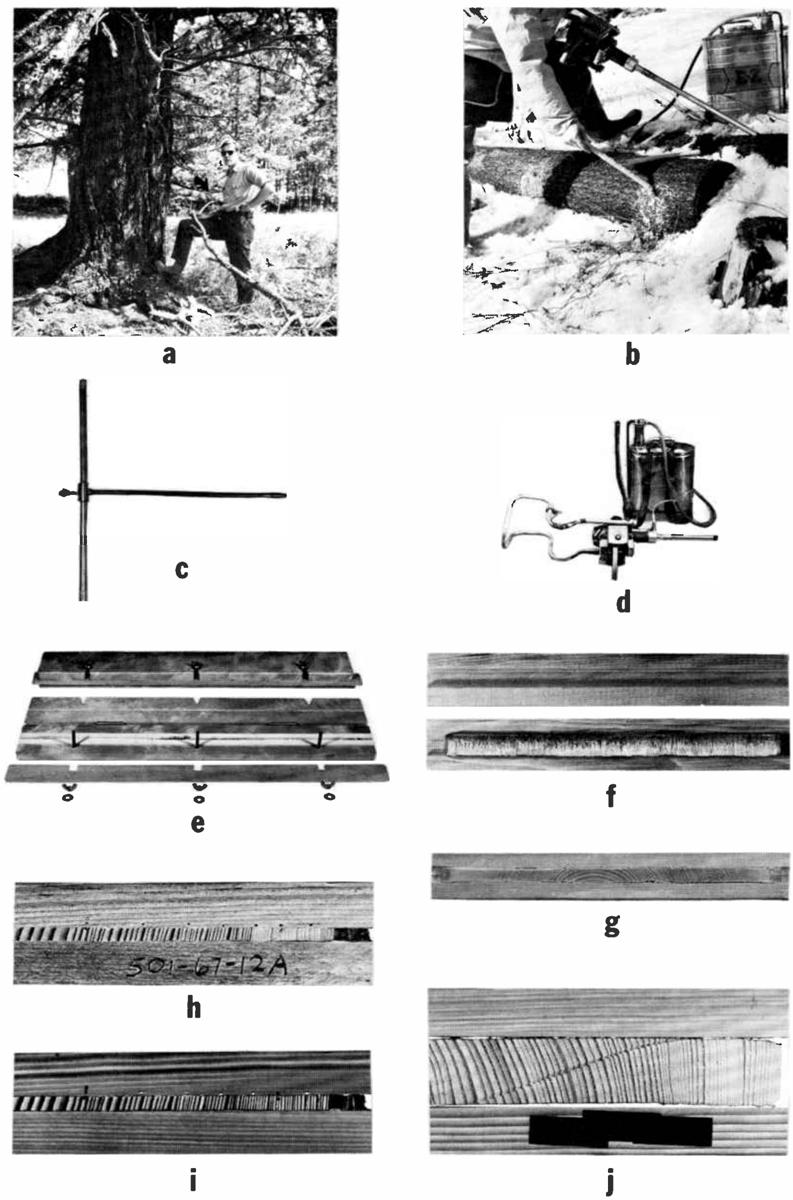

Fig. 1. Increment cores. *a*, extracting 5 mm diameter core with 40 inch increment borer (arrow); *b* taking 3/4 inch diameter cores with G. S. C. power-driven increment borer; *c*, Swedish increment borer (handle 18 inches long); *d*, G. S. C. power-driven increment borer (overall length 48 inches as shown); *e*, small core mounting jogs (26 inches long); *f*, 3/4 inch diameter core and core mount; *g*, mounted and sanded 3/4 inch core; *h*, photograph of portion of 5 mm diameter core; *i*, X-ray positive of same 5 mm core; *j*, X-ray positive of portion of mounted 3/4 inch diameter core.

Many factors can affect the quality of the radiographs from field sampling and specimen preparation to X-ray exposure and film development. The problems encountered in producing X-ray negatives of dendrochronological specimens are discussed and conclusions concerning some of the advantages of the X-ray technique are presented.

Specimen Preparation and X-Ray Examples

Preparation of Increment Cores

Two types of increment cores are used for tree-ring studies at the Geological Survey of Canada (Fig. 1). The small cores (4.5 or 5 mm. diameter) are extracted with Swedish increment borers, ranging from 12 to 40 inches in length. The large cores (5/8 or 3/4 inch diameter) are taken with a powerdriven increment borer developed at the Geological Survey with the help of G. A. Meilleur.

The small increment cores are allowed to dry and mounted with resin glue between two mounting sticks that are 1/2 inch wide and 3/32 inch thick. These mounting sticks are grooved on one edge to accommodate the cylindrically-shaped core. A mounting jig has been designed to hold the cores and their mounting sticks in the proper position while the glue is drying. The cores are mounted with the long axis of the longitudinal tracheids in a vertical position. The rounded portions of the cores projecting above and below the core mounts are sanded with successively finer grits of sandpaper until this transverse cross section of the core has a polished surface and is flush with the top and bottom surfaces of the core mounts. This technique provides a core of uniform thickness and with vertically oriented cells, suitable for X-ray exposure, and with two sanded surfaces that may be examined under the microscope for dating and ring-width measurement. Fragmentary cores can be reconstructed and held together for X-ray processing by this mounting technique.

The large cores are completely incased between two mounting sticks two inches wide and one inch thick. Each mounting stick has a groove of the proper shape and depth to accommodate one half of the core. Resin glue is used in the grooves (around the core) and between the seams fromed where the mounts are in contact. The long axis of the longitudinal tracheids is aligned parallel to this seam. The core mounts are held together firmly with C-clamps until the glue is dry. A 3/32 inch thick transverse cross section of the core is produced by making two parallel saw cuts with a circular table saw or a band saw. These saw cuts are made along the longitudinal axis of the core mounts and perpendicular to the seams between the mounts. This technique produces three mounted core components, two of which can be sanded and examined under the microscope for dating and ring-width measurement, and one with proper cell alignment and thickness for X-ray exposure. Each component has wood grain running in two different directions which prevents the mounted specimen from warping.

Preparation of Wood Cross Sections

Transverse cross sections of tree trunks or wood fragments (Fig. 2) are prepared for X-ray exposure by making two parallel saw cuts through the specimen perpendicular to the long axis of the longitudinal tracheid cells. These sections are cut 3/32 inch thick. If the sample is fragile, a single layer of backing tape is placed on the plane surface formed by the first saw cut before the second cut is made. The backing tape is retained during X-ray exposure. No surface preparation, other than the rough saw cut, is required.

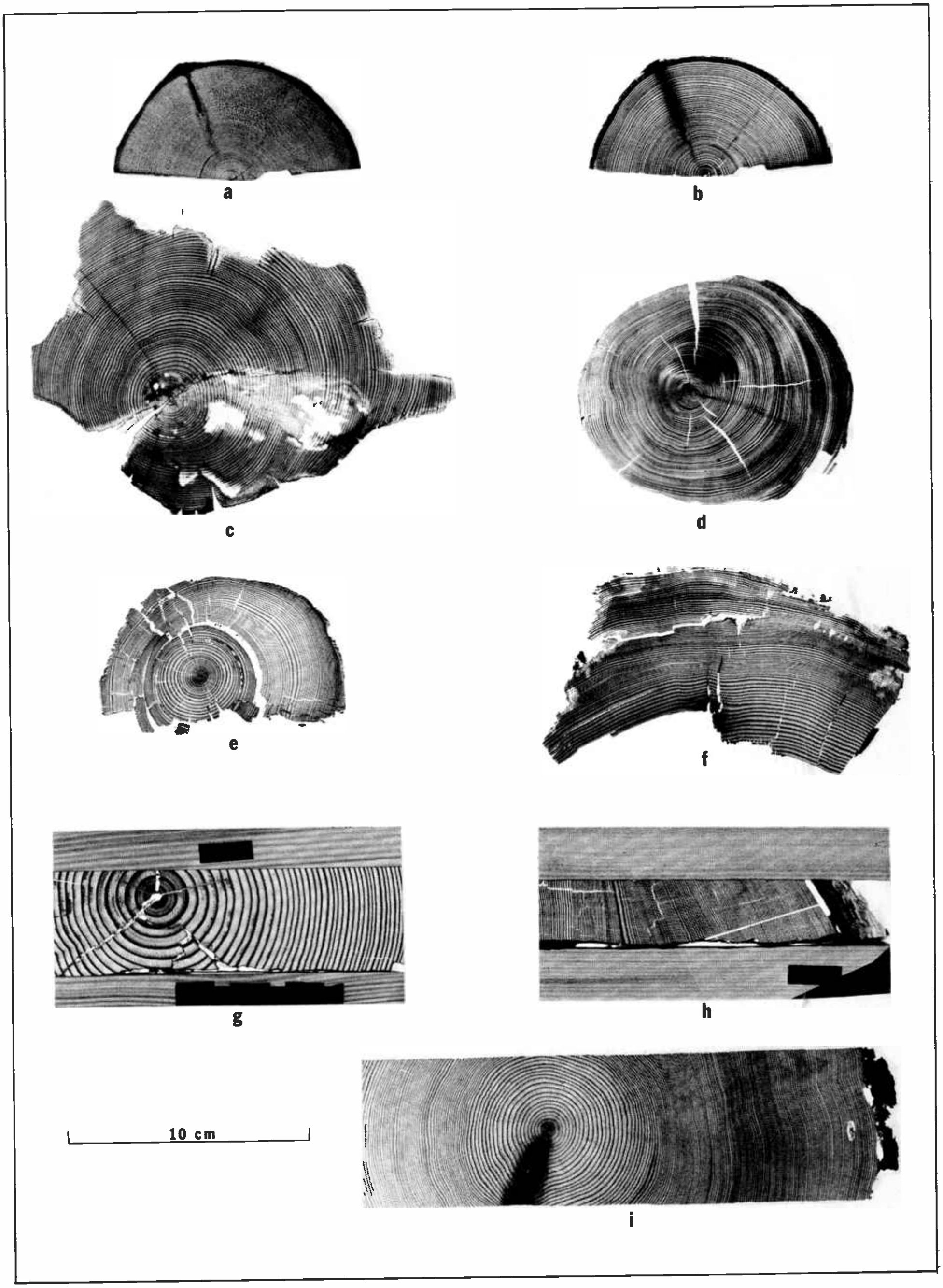

Fig. 2. Cross sections. *a*, photograph of cross section prepared for X-ray exposure; *b*, X-ray positive of *a* (*c* through *i* are also X-ray positives); *c*, archaeological specimen; *d*, interglacial specimen (greater than 54,000 years old); *e* and *f*, fragile submerged forest sections with backing tape; *g* and *h*, V-cut stump sections with mounting boards; *i*, section from block of manageable size from tree disc. All sections are 3/32 inch thick.

Wedge-shaped slabs of wood cut from the flat surface of tree stumps ("stump V-cuts") are prepared for X-ray exposure in the following manner: (1) a flat surface, parallel to the long axis of the longitudinal tracheids, is chiseled along one edge of the V-cut slab, (2) a 1/2 inch thick mounting board is glued to this chiseled surface, (3) the specimen is "squared off" by making 90° saw cuts along the top of the slab, along the outer edge of the side opposite the first mounting board, and along the bottom of the specimen, (4) a second mounting board is glued to the plane surface opposite the first mounting board, and (5) 3/32 inch thick transverse cross sections of the tree-ring specimen are produced by making saw cuts along the long axis of the mounting boards.

In order to produce samples of manageable size from very large tree trunk discs, blocks containing the entire tree-ring series are cut and mounted in a manner similar to that described for the V-cut stump samples.

Charcoal

Many archaeological tree-ring specimens are in the form of charcoal, creating a need for a method of X-raying charcoal if density measurements are required. Experiments have been conducted on techniques of X-raying both coniferous and hardwood samples, and good quality X-ray negatives have been produced (Fig. 3). The method is as follows: (1) a transverse cut is made through the specimen with a band saw in the area that contains the outermost rings, (2) extraneous particles are removed with compressed air from the plane surface formed by the saw cut, (3) a single layer of backing tape is applied to this saw-cut surface, (4) a 3/32 inch thick cross section is obtained by making a second saw cut through the specimen parallel to the first cut, (5) X-ray film is exposed through this thin cross section.

Tree-Ring Characteristics

The amount of information to be obtained from tree-ring series is greatly increased if intra-annual ring density data are used to supplement tree-ring width data. However, new problems are introduced regarding methods of preparation and analysis. Tree-ring characteristics such as false rings, frost rings, faint latewood, microscopic rings, locally absent rings, partially formed rings, and compression wood can be presented graphically and quantitatively by X-ray and densitometric techniques. Radiographs of some of these tree-ring characteristics (Fig. 4) demonstrate the quality of information that can be obtained by the X-ray technique, and suggest the added complexities this method may introduce.

X-Ray Technique

Techniques for producing the X-ray negatives of tree-ring samples have been developed at the Nondestructive Testing Laboratory, Mines Branch (Fig. 5). Research by Polge (1965a, 1965b, 1966) has been a useful guide.

X-Ray Apparatus

The X-ray apparatus used is a Picker portable instrument with a 50 kilovolt (maximum) capacity, 1.5 mm focal spot, .5 mm thick beryllium window, and self-rectifying circuit. It operates on a 110 volt, 60 cycle power source, and has an air-cooled oil circulation system. The duty cycle at 50 kilovolts is 50 percent at 10 milliamperes and 100 percent at 7 milliamperes. Soft X-rays are produced by this machine.

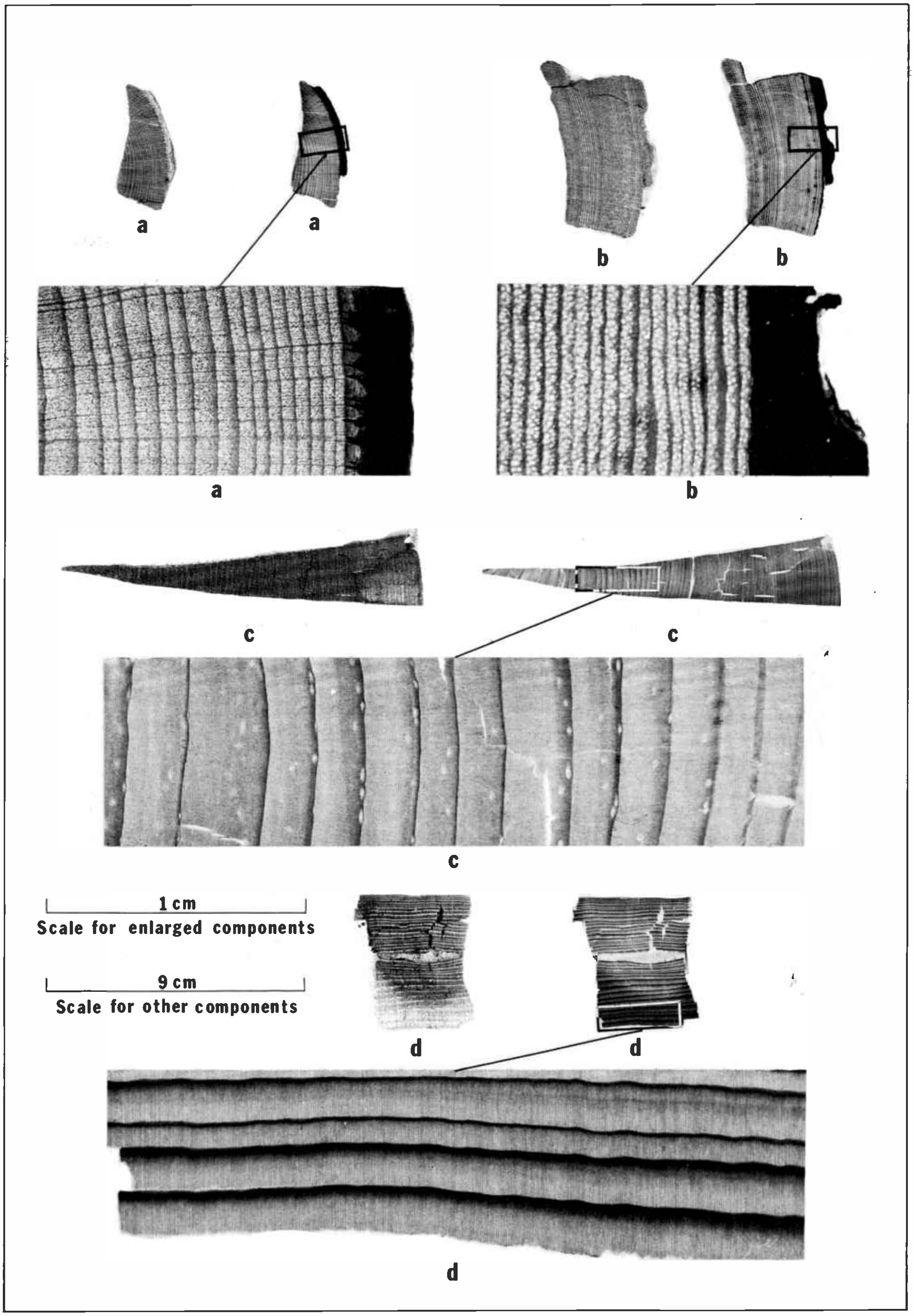

Fig. 3. Charcoal cross sections *a* and *b* are sections from broad-leaved trees; *c* and *d* are sections from conifers. Each group of three components includes a photograph of a charcoal specimen (left), and X-ray positive of the same specimen (right), and an enlargement of a portion of the X-ray positive (below other two).

Stationary Technique

One method for producing the radiographs is to hold the X-ray source, tree-ring specimen, and film stationary during exposure. The wood sample is placed on single emulsion radiographic film and subjected to X-radiation from a distance of 48 inches and an angle of 90°. Under normal conditions the exposure is for 2 minutes at 22 kilovolts and 5 milliamperes.

In-Motion Technique

Proper definition of annual ring density is obtained only if the X-rays penetrate the sample at an angle parallel to the long axis of the longitudinal tracheids. A technique has been devised to expose long tree-ring samples at the desired angle by in-motion radiography. X-radiation is transmitted through a 1 mm wide slit from a stationary source 10 inches from the tree-ring specimen positioned on the film. The specimen and film are supported by a carriage moving at a slow and uniform speed 3/8 inch beneath the slit. Successful experiments have been conducted using a carriage speed of 2 inches per minute with the X-ray machine set for 22 kilovolts and 6.5 milliamperes.

Film

Experiments have been conducted using double emulsion radiographic film, single emulsion radiographic film, and photographic film. If the double emulsion film is exposed at an angle other than 90° a double image will result from thy parallax effect, limiting the use of this type of film for some purposes. A fine grain photographic film was used with good results. Photographic film can be purchased in continuous rolls in bulk quantities.

The film selected for standard use is a single emulsion radiographic film with very high contrast and very fine grain (Kodak X-Ray Film, Type R). Tests were conducted by varying the kilovoltage and milliamperage until the desired contrast was obtained by using 22 kilovolts and 5 milliamperes. All exposures have been made on film in sealed, opaque, paper envelopes. Kodak liquid developer and replenisher for industrial radiography has been used at the recommended development time of 8 minutes at 68°F.

Discussion

The concept of using X-ray and densitometric techniques in tree-ring studies is relatively new. A great deal remains to be determined concerning the most useful methods of preparation and the many applications of the principle. The objective of our investigations, to this point, has been to make a general evaluation of the advantages of the X-ray technique and to examine the factors affecting the quality of X-ray negatives to be used for crossdating and for climatic compairsons. *Optical density* measurements of the X-ray negatives have been used, but techniques to use *wood density* measurements are now being investigated.

Advantages of the X-ray Technique

In terms of dendrochronological requirements, the technique of producing X-ray negatives of tree-ring samples has certain advantages over the use of sanded wood surfaces, microtome thin sections, or photographs of the specimens. The X-ray technique is nondestructive and produces a permanent and exact scale record of ring density. Both ring-width and ring-density data can be extracted from radiographs. This information can

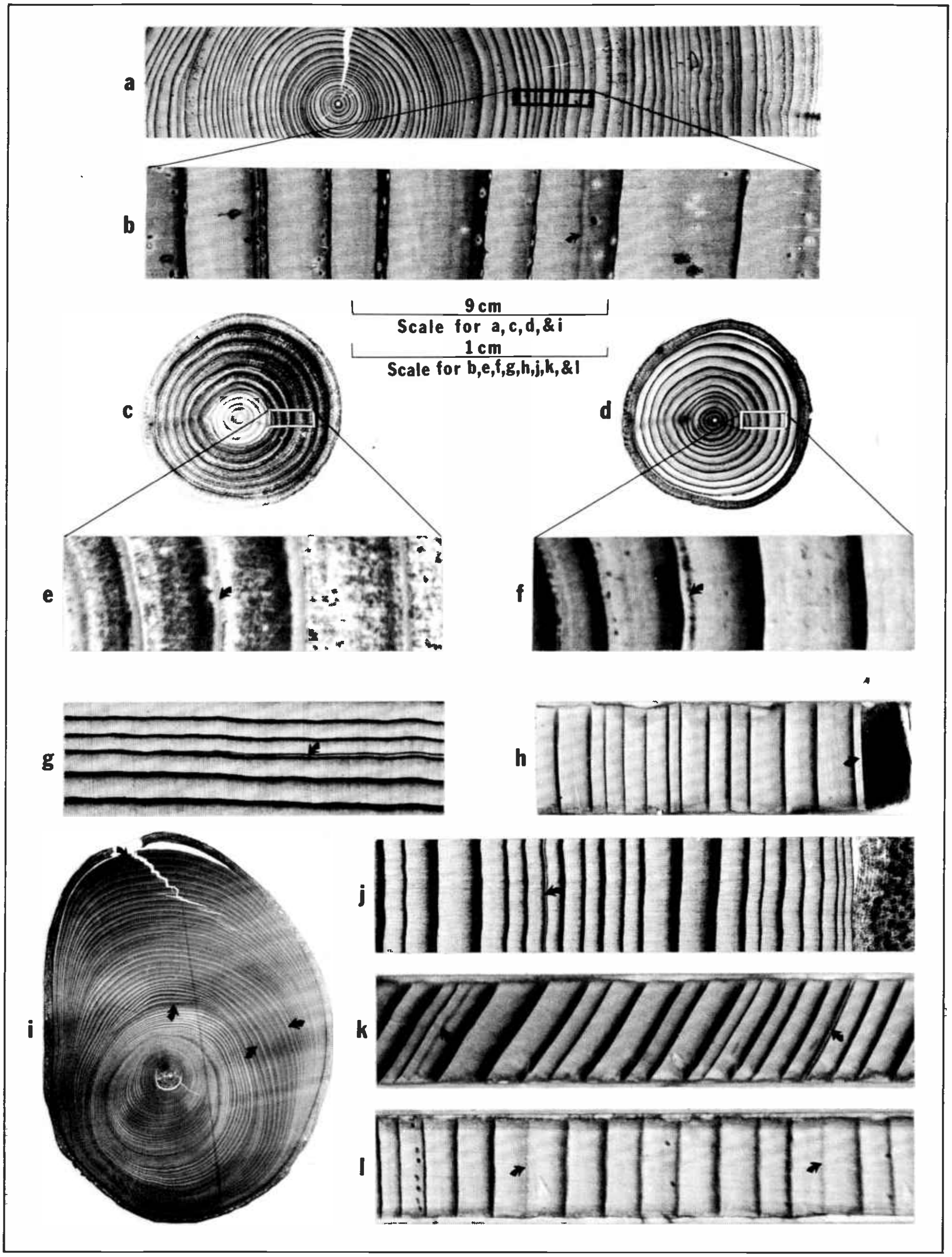

Fig. 4. Tree-ring characteristics. All 12 components are X-ray positives. *a*, tree-ring section; *b*, enlargement of *a* showing false rings (doubles): latewood double (left arrow) and earlywood double (right arrow); *c* and *d*, two forms of same cross section (*c*, green state and *d* dry state); *e* and *f*, enlargements of *c* and *d* showing frost ring (arrows); *g*, locally absent ring; *h*, incompete ring (core taken during growing season); *i*, compression wood (general area between arrows); *j*, microscopic ring; *k*, false ring (left arrow) and microscopic ring (right arrow); *l*, faint latewood (both arrows).

be quantitatively measured and presented graphically in the form of density plots (Jones and Parker 1970) and as photographic prints made from the X-ray negatives. Quantitative measurements of a number of intra-ring features such as frost rings, false annual rings, and latewood density can be made. Relatively little time is required to prepare the sample for X-raying and to make the radiograph. Much longer tree-ring series can be recorded on a single X-ray negative than can be prepared on a single microtome thin section. Well defined ring series can be produced by X-raying charcoal, and radiographs of very rotten wood will produce images sufficient for ring-width measurement. Color distortions such as stains and heartwood and sapwood differences do not affect the radiograph as they do photographic negatives or other media using reflected light. Intense and easily controlled transmitted light can be projected through the radiograph on a densitometer to record the density information.

In a few ways, however, the X-ray technique is less useful for tree-ring analysis than the others mentioned, and should supplement rather than replace these other methods. Some aspects of wood cell structure can be observed from good quality radiographs but microtome thin sections are much more useful for examination of microscopic features. Very small rings can be observed more accurately for ring-width measurement on finely sanded surfaces of tree-ring samples than on radiographs, *unless* the angle of X-radiation exposure is exactly parallel to the long axis of the longitudinal trachieds. If the angle is correct, however, very fine ring definition is obtained by the X-ray technique. Photographs also have certain advantages over radiographs for illustrative purposes or for color differentiation.

Factors Affecting X-Ray Quality

The quality of X-ray negatives to be used for dendrochronological purposes can be affected by a large number of factors (Fig. 6). These factors are related to the condition of the specimens, methods of specimen preparation, and X-ray and film developing techniques. One of the most important considerations is the alignment of the xylem cells within the specimen to be X-rayed. Increment cores should be taken at an angle perpendicular to the longitudinal tracheids if density studies are to be made. If X-ray negatives are produced by radiation penetrating the specimen at some angle other than parallel to the long axis of the longitudinal tracheids, the ring boundaries are poorly defined and the density measurements are distorted. The angle of X-ray exposure of the core can be changed by tipping the X-ray apparatus or the core and film to compensate for this condition, but this procedure is time consuming and usually can be avoided if the cores are correctly extracted.

There are other factors related to the condition of the specimen and preparation techniques that will affect quality such as the presence of foreign matter (preservatives, glue, sand, etc.), root flare, branch distortion, cracks, state of preservation (rotten wood), inherent dendrochronological quality of the species, moisture content (green or dry state), size of rings, thickness of specimen, variations in thickness, and ring boundary alignment.

Many factors related to X-ray and film development also can affect quality. Contrast and optical resolution are affected by kilovoltage, milliamperage, slit size, X-ray window (filter), source to film distance, source to specimen distance, specimen to film distance, type of film, development of film, and exposure time.

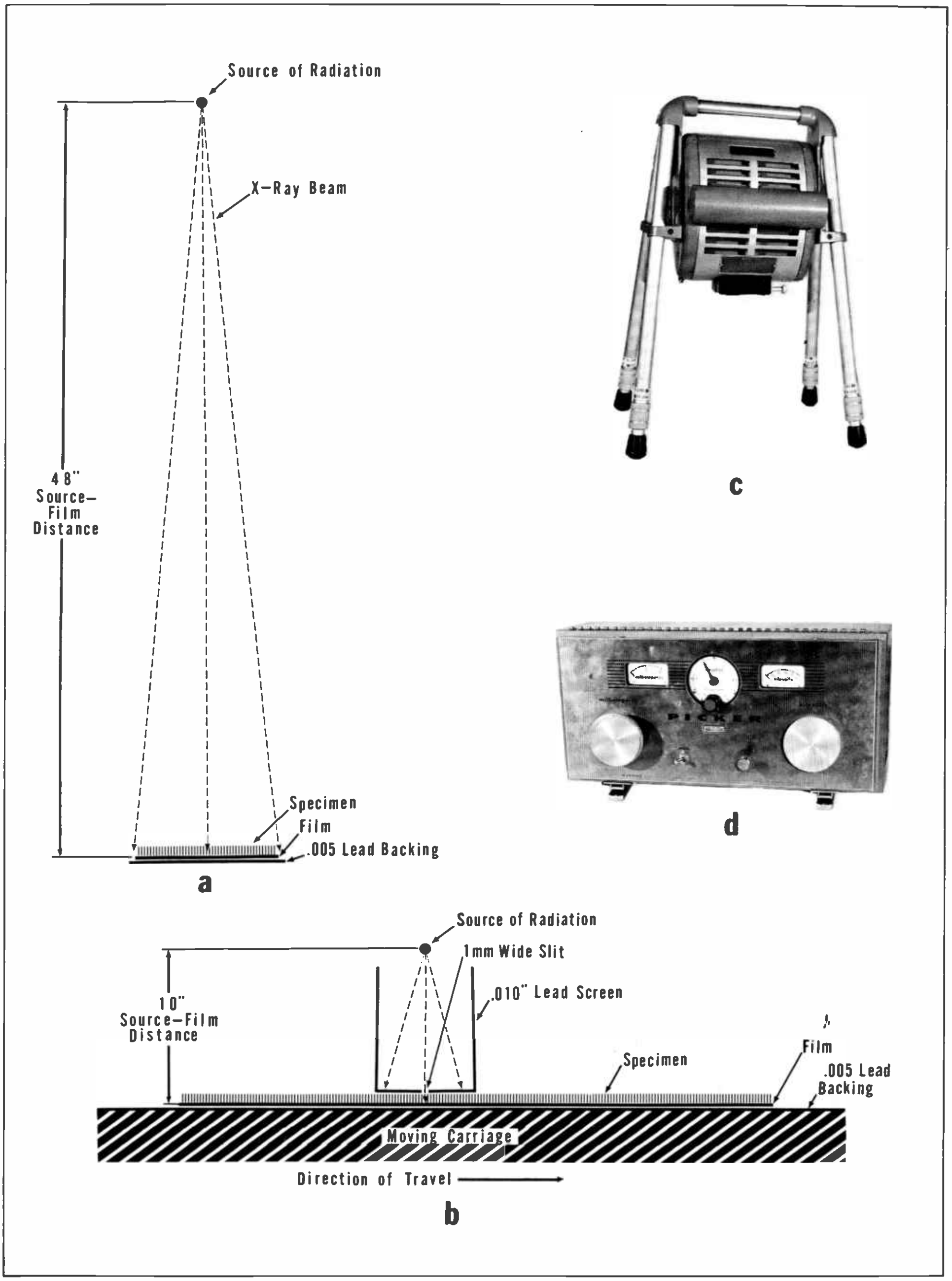

Fig. 5. X-ray techniques *a*, stationary technique; *b*, in-motion technique; *c* X-ray tube; *d*, electronic controls.

Conclusion

The potential for using the X-ray technique for dendrochronological analysis has just begun to be investigated. Many technical problems remain to be resolved, but the quantity and quality of information to be obtained from density studies makes this new approach to tree-ring studies well worth while.

Acknowledgements

We wish to acknowledge the help of A. Marleau in the development of X-ray methods, D. E. Field and E. Fitzpatrick for assistance in specimen preparation techniques, F. J. Cooke for the photography, and J. A. Heginbottom for editorial suggestions. Advice from H. Polge and H. V. Green also has been most useful. We also acknowledge the Nondestructive Testing Section, Physical Metallurgy Division, Mines Branch for the radiography of the specimens.

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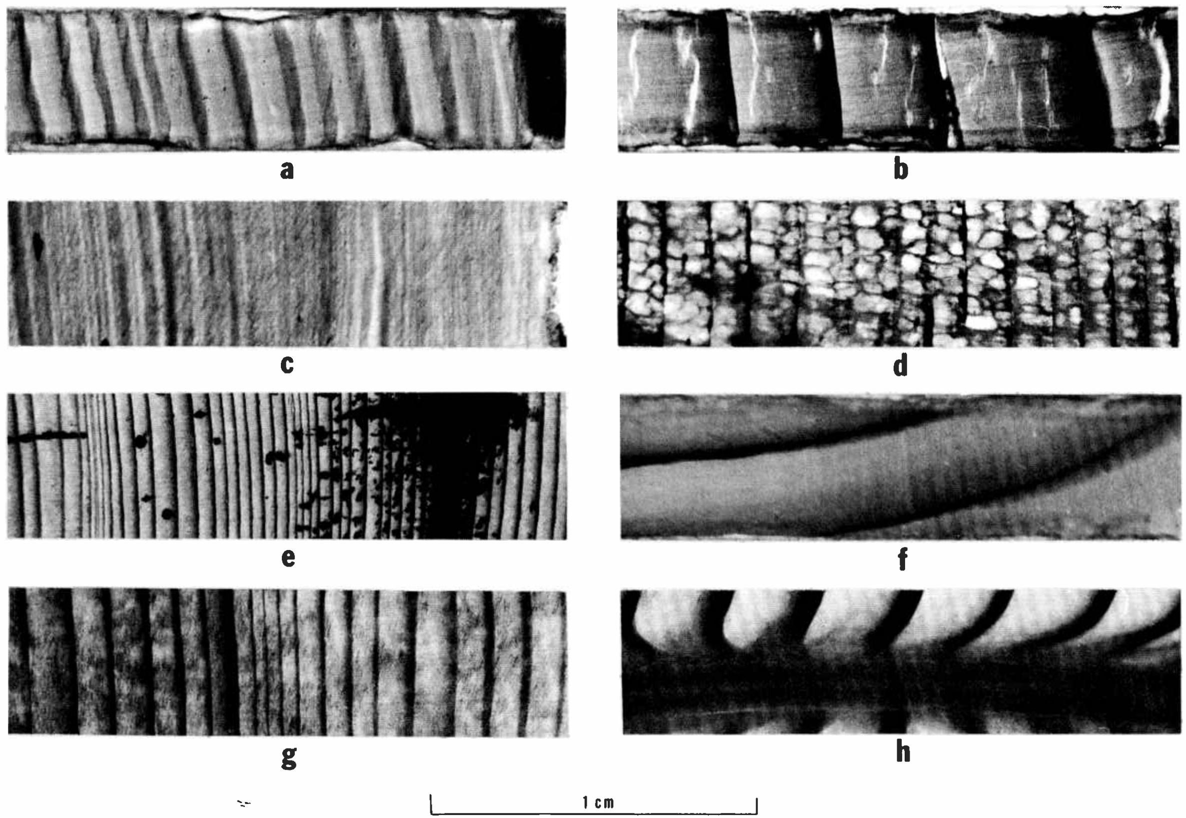

Fig. 6. Some factors affecting X-ray quality. All eight components are X-ray positives. *a*, misaligned xylem cells (large rings); *b*, cracks; *c* missaligned xylem cells (small rings); *d*, rotten wood (ring widths are discernible but density is distorted); *e*, foreign matter (soil particles); *f*, misaligned rings (core taken at incorrect radial direction); *g*, foreign matter (polyethylene glycol preservative); *h*, limb distortion.