sample. The platinum wire should be wrapped so that the sample is supported throughout the entire combustion; carbon particles are formed if any of the sample drops to the bottom of the flask before burning is complete. A portion of the sample or a piece of low-ash filter paper is used for the ignition wick as shown in Figure 1. To increase the absorption of radiant energy, the tip of the wick is blackened with lead pencil graphite. The sample holder is hooked over the positioning rod, lowered into the flask, and the sample height adjusted so that the wick is in line with and directly above the projector bulb. Acid, alkali, or water is added to the bottom of the flask to dissolve the ash after combustion. The balloon and adapter are removed, and the flask is flushed and filled with oxygen gas. After the balloon is replaced and a safety shield is in position, the lamp is turned on until combustion is started. As soon as combustion is complete, the platinum wire is dropped to the bottom of the flask in contact with the ash solvent. The flask, which may be cooled rapidly by spraying the outer surface with water, is held until any aerosol disperses; then the ash solution and the water used to rinse the balloon and the combustion flask are aspirated into a flask for analysis.

RESULTS

The apparatus has been used for the analysis of iron, copper, and chloride in woodpulp. Some of the iron recovery data are presented in Table I.

Iron in the form of ferric chloride solution was added to woodpulp and Whatman No. 40 filter paper. The ash was dissolved in 20 ml. of 1:1 HCl, and the iron determined with 4,7-diphenyl-1,10-phenanthroline (bathophenanthroline) using a modification of the procedure of Gahler, Hamner, and Shubert (*2*).

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Rapid Scanning of Radioactive Thin Layer Chromatograms

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A SIMPLE, convenient method is available by which radioactivity on thin layer chromatoplates (TLC plates) can be scanned rapidly. Instruments which routinely scan paper chromatograms can be used to scan thin layer plates, provided the plate dimensions are compatible with the slot dimensions of the particular instrument. An inexpensive technique for coating plates with a wide range of dimensions has been described (*2*).

EXPERIMENTAL

In order to utilize paper chromatographic scanning techniques for TLC plates, a Tracerlab SC 55 chromatogram scanner (Figure 1) was employed. The scanner consists of a sample changer, an end-window Geiger tube, a ratemeter, and a recorder. In operation, the strip is taped to the aluminum slide of the scanner, and the slide is pulled by a pinion and rack gear which is driven from a flexible shaft coupled to the recorder chart drive. The scanning speed equals the chart drive speed. Therefore, at the completion of the scan, the chromatogram strip can be laid alongside the recorder trace for direct identification of each location of radioactivity. A Mylar film, 0.9 mg. per sq. cm., serves as the window of the Geiger tube which is at a distance of 0.26 inch from the coated TLC plate.

To coat the thin glass plates required for the scanner, narrow strips of masking tape were applied to opposite edges of ordinary window glass (46 mm. × 200 mm. × $^3/_{32}$ inch) (*2*). The tape extended over the plate, fastening to the bench top covered with paper toweling. An aqueous slurry of Anasil B (Analabs, Hamden 18, Conn.) was spread by sliding a glass rod across the plate, with the thickness of the tape determining the thicknesses of coating.

After spotting and developing the chromatogram in the usual way, the chromatoplate was taped to the slide of the scanner and a tracing obtained. Figures 2 and 3 are the tracings which resulted from scanning the chromatoplates obtained with oleic acid-1-C^{14} and triolein-1,1′,1″-C^{14} (Tracerlab). Figure 4 is a tracing of the triolein-1,1′,1″-C^{14} chromatoplate before final purification. Linoleic acid-1-C^{14} moved the same distance as the oleic acid-1-C^{14}.

DISCUSSION

The method described provides a rapid and simple way to analyze results obtained from TLC plates of tagged

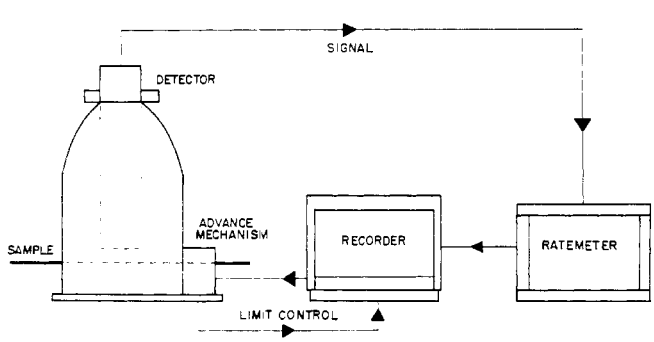

Figure 1. Diagram of continuous scanning system

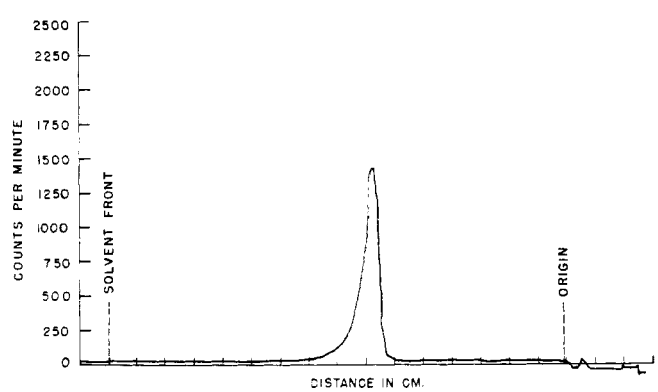

Figure 2. Tracing of a chromatoplate of oleic acid-1-C^{14} (Tracerlab)

Adsorbent. Anasil B (Analabs)
Solvent. Ligroin–diethylether–acetic acid, 20/80/1 v./v./v.
Development time. 27 minutes
Full scale. 2500 c.p.m.

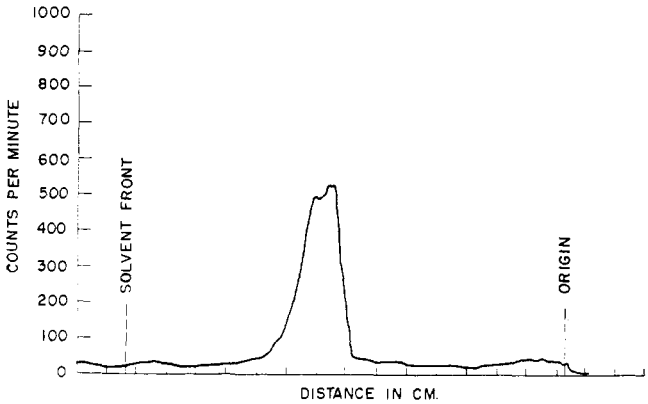

Figure 3. Tracing of a chromatoplate of triolein-1,1′,1″-C^{14} (Tracerlab)

Adsorbent. Anasil B (Analabs)
Solvent. Ligroin–diethylether–acetic acid, 90/10/1 v./v./v.
Development time. 50 minutes
Full scale. 1000 c.p.m.

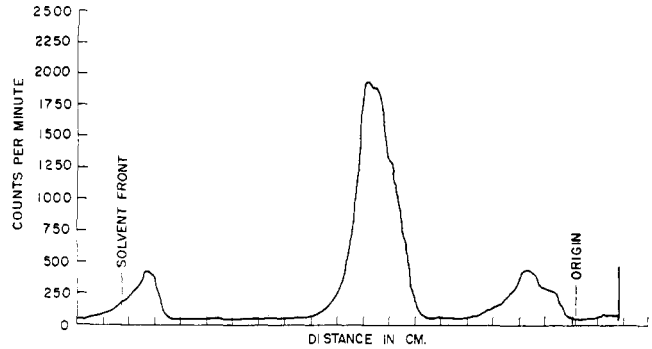

Figure 4. Tracing of a chromatoplate of triolein-1,1′,1″-C^{14} before purification

Adsorbent. Anasil B (Analabs)
Solvent. Ligroin–diethylether–acetic acid, 90/10/1 v./v./v.
Development time. 35 minutes
Full scale. 2500 c.p.m.

compounds. Thus it is a valuable adjunct to other proven methods which serve the same function such as autoradiography (*3*) and liquid scintillation counting (*1*, *4*) of isolated chromatoplate sections.

Depending on the coating thickness and the slit width and time constant of the scanner, the efficiency was about 2%. With more recent developments, including thinner windows, this should be markedly increased. A step chromatogram scanner can be used instead of the continuous scanner described here. This will give a printed record of the counts of each segment and can be adjusted with preset time and preset counts to give great accuracy even with minimum quantities of radioactivity.

The scanning method should prove particularly helpful in cases where rapid analysis is essential, as with compounds containing short-lived isotopes—i.e., sulfur-35 and iodine-131. Any laboratory working with radioactivity and having a chromatogram scanner with a suitable slide can, with almost no additional expense, extend its capabilities to TLC, since no modification of the scanning instrument is necessary. In addition, the TLC results are easily interpretable in terms generally used with radioactivity.

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Preparation of Deuterated Solvents for Nuclear Magnetic Resonance Spectrometry

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Deuterated chloroform and acetone are useful and sometimes indispensable solvents for studies involving proton nuclear magnetic resonance spectrometry. Unfortunately, these solvents are relatively expensive for routine use and it is common procedure either to use minimal volumes or recover the solvents for reuse. It should be noted, however, that 100 grams of deuterated chloroform, which costs approximately $100, contains only 40 cents worth of deuterium based on the cost of heavy water at $60 per kilogram (in 125-pound lots). Deuterated chloroform can be produced at high efficiency from heavy water by the following reaction

$$CCl_3COCCl_3 + D_2O \xrightarrow{\text{pyridine}} 2CDCl_3 + CO_2$$

PROCEDURE

Two hundred and sixty-five grams of hexachloroacetone (one mole), 40 ml. of heavy water (2 moles-100% excess), and 10 ml. of pyridine were added to a distillation flask. Two phases formed. On slow heating, a mixture of $CDCl_3$, D_2O, and pyridine distilled out as the reaction proceeded. The $CDCl_3$ was purified by a second distillation, dried over calcium sulfate, and redistilled. One hundred and ninety grams of deuterated chloroform were obtained at a cost of approximately five cents a gram. The chloroform should be either stored under refrigeration to prevent decomposition, or stabilized with a small quantity of deuterated ethanol.

The reaction chosen to prepare the deuterated acetone was as follows.

$$CH_3COCH_3 + \text{excess } D_2O \xrightarrow{\text{LiOD}} CD_3COCD_3 + 6HDO$$

PROCEDURE

Fifty milliliters of analytical reagent grade acetone, 100 ml. of 99.7% deuterated heavy water, and 0.4 ml. of a saturated solution of LiOD in D_2O are mixed together. The LiOD solution can be prepared by reacting lithium wire with D_2O until LiOD begins to precipitate. The mixture is left standing for 30 minutes. The acetone is then separated by distillation and the process is repeated using new 100-ml. portions of heavy water until the acetone has undergone four exchanges. The heavy water samples left from the first preparation can be reused with a new batch of acetone, and a fifth sample of heavy water added at the end of the process to yield the same high purity deuterated acetone. By extending the process, and limiting the number of distillations to five, (discarding the heavy water with the highest hydrogen content after each preparation) the materials cost of one batch of deuterated acetone equals the cost of 100 ml. of heavy water or about 20 cents per gram of deuterated acetone. This process has been scaled up by a factor of 10 in our laboratory.

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