Routine analytical methods have been developed for characterizing Bayer liquor. The methods consist of instrumental techniques, including gas chromatography for the low and intermediate molecular weight compounds and gel permeation chromatography for the high molecular weight material. Gas chromatography/mass spectrometry was used to identify several compounds in addition to those previously reported in the literature. Two spent liquor samples, together with corresponding lake water samples, were characterized to demonstrate applicability of the methods. Results of the analyses provided information for better understanding the nature of Bayer liquor.
mixture was vigorously shaken for 2 minutes. The sample was heated for 1 hour at approximately 70°C in a water bath. After cooling, the butanol layer was removed with a disposable Pasteur pipet, without removing any of the aqueous phase, and 1 μl of the solvent containing the butyl esters was injected into the GC.

To prepare a lake water sample for analysis, 20 ml was placed in a Petri dish and allowed to evaporate in a hood. One milliliter of 12% sodium hydroxide was used to rinse the air dried material into a 24 ml vial containing ~15 mg of nonanoic acid as an internal standard. The remainder of the sample preparation procedure was the same as described for low molecular weight compounds in a liquor sample.

Samples were run on a Hewlett-Packard 5880 GC equipped with a flame ionization detector. The column was 1/8 in. O.D. x 10 ft. stainless steel packed with 10% SP-1000 on 100/120 mesh Chromosorb WAW (Supelco, Inc.). Chromatographic conditions were the following:

- **Injection port temp.** 250°C
- **Detector temp.** 350°C
- **Initial oven temp.** 100°C
- **Time at initial temp.** 5 min.
- **Program rate** 10°/min.
- **Final oven temp.** 250°C
- **Time at final temp.** 5 min.
- **Carrier flow** 30 ml/min.
- **Carrier** Helium
- **Attenuation** X3/32
- **Chart speed** 0.5 cm/min.

A typical chromatogram of the low molecular weight compounds is shown in Figure 1. The compounds were quantified using an internal standard technique.

Organic acids were named as carboxylic acids, even though the compounds actually analyzed were butyl esters, silyl derivatives or a mixture of both, depending on the particular technique used. However, these compounds exist as sodium salts in the liquor.

**Intermediate Molecular Weight Compounds**

The liquor sample preparation procedure outlined for low molecular weight compounds was followed down to removal of the butanol layer from the 24 ml vial. At this point, the butanol layer was placed in a 30 ml beaker and allowed to evaporate overnight at room temperature in a hood. One milliliter of TRI-SIL silyl derivatizing agent (Pierce Chemical Company) was added to the dried sample. The sample and TRI-SIL were mixed, transferred to a 4 ml screw cap vial and centrifuged. One microliter of derivatized sample was injected into the GC.

To prepare a lake water sample for analysis, 20 ml was placed in a Petri dish and allowed to evaporate in a hood. One milliliter of 12% sodium hydroxide was used to rinse the dried material into a 24 ml vial. The remainder of the sample preparation procedure was the same as described for intermediate molecular weight compounds in a liquor sample.

Samples were run on a Hewlett-Packard 5880 GC equipped with a capillary column inlet and a flame ionization detector. The column was a 0.25 mm I.D. x 30 m DB-5 fused silica capillary (J&W Scientific, Inc.). Chromatographic conditions were the following:

- **Injection port temp.** 300°C
- **Detector temp.** 350°C
- **Initial oven temp.** 50°C
- **Final oven temp.** 325°C
- **Program rate** 10°/min.
- **Time at final temp.** 5 min.
- **Carrier flow** 30 cm/sec.
- **Carrier** Helium
- **Attenuation** X3/32
- **Chart speed** 1 cm/min.

A typical capillary GC chromatogram is shown in Figure 2.