PLAINTIFF'S EXHIBIT

DOW-720

ANALYTICAL REPORT

MIDLAND ANALYTICAL LABORATORIES

DOW CHEMICAL U.S.A.

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TITLE DETERMINATION OF ASBESTOS IN CELL EFFLUENT USING ATOMIC ABSORPTION SPECTROSCOPY

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Introduction

Asbestos levels in our chlor-alkali process are being determined by a time consuming electron microscopic technique. H. A. Chagnard, Louisiana Division, has reported that an elemental analysis for magnesium by atomic absorption spectroscopy can be used to determine asbestos levels in cell effluent. Since a rapid analysis would be desirable for evaluating filtration experiments for asbestos removal from cell effluent, we have evaluated this atomic absorption procedure.

Safety

Cell effluent is a corrosive alkali and hydrochloric and hydrofluoric are corrosive acids. Handling of these materials require the use of eye goggles and protective clothing. Hydrofluoric acid is especially corrosive to tissue and contact with the skin should definitely be avoided at all times.

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Experimental

Three samples each of unfiltered and filtered cell effluent were obtained from the Analytical Laboratories Microscopy Group. Asbestos contents of all had been determined using an electron microscope coupled with an X-ray fluorescence analyzer. The apparent asbestos content of these samples was then determined with slight modification of the atomic absorption method developed by Chagnard. A detailed description of the method used is as follows.

Determination of Asbestos in Cell Effluent

1. Shake sample vigorously, and immediately filter a 100 ml portion of sample through a 47 mm Metricel* 0.45 micron filter. A Millipore* membrane cell filter holder may be used for these filtrations. This holder or a similar filter holder must be used with a vacuum flask to speed up acid washes. Larger portions are recommended for samples of low asbestos content to increase sensitivity.

2. Wash filter rapidly with 3, 50 ml portions of 0.1 N HCl. (Each portion of acid should pass through the filter in less than ten seconds to minimize asbestos losses.)

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3. Place the filter disc in the bottom of a 150 ml polyethylene beaker and add 1.0 ml of 48% reagent grade or 55% technical grade hydrofluoric acid. Allow the filter to stand for 30 minutes to dissolve the silica compounds.

4. Add 25 ml of 5% boric acid to the beaker. Swirl the beaker and place it into a shallow water bath about 50°C. Allow to stand 15 minutes to inactivate the excess hydrofluoric acid.

5. Add 74 ml of distilled water to the beaker.

6. Repeat above procedure without a sample to obtain a reagent blank.

7. Obtain atomic absorption data using a Perkin Elmer 303 atomic absorption unit (or equivalent) using the 285 mμ magnesium wavelength and an air-acetylene flame. A standard curve should be produced with 0, 0.5, 1.0 and 2.0 micrograms Mg per milliliter standard solutions. This curve will deviate slightly from linearity above 2.0 μg Mg/ml. The standards should contain the same amount of boric acid and hydrofluoric acid as the samples.

8. Calculate ppm asbestos by multiplying μg Mg/ml in sample by 3.83 (factor for chrysotile asbestos with molecular formula $3MgO \cdot 2SiO_2 \cdot 2H_2O$).

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Results

Table I compares the data obtained by the microscopic method with those obtained by atomic absorption. For the microscopic data, the micrograms/milliliter (μg/ml) values were calculated from the million fibers/liter data (MFL). These μg/ml data should be used only for comparison purposes since they are based on an average length-average diameter-known density calculation.

Table I - Comparison of Microscopic and Atomic Absorption Measurements of Asbestos in Cell Effluent

Sample	Asbestos-Microscopy		Asbestos-Atomic Absorption[a]
Unfiltered	MFL	μg/ml	μg/ml
10/23/74	46,000	0.66	4.4
10/24/74	35,000	0.60	4.1
10/29/74	>50,000	>1.15	2.3
Filtered			
10/22/74	96	0.0005	0.015 ± 0.007[b]
10/24/74	260	0.0004	<0.02
10/29/74	351	0.0031	<0.04

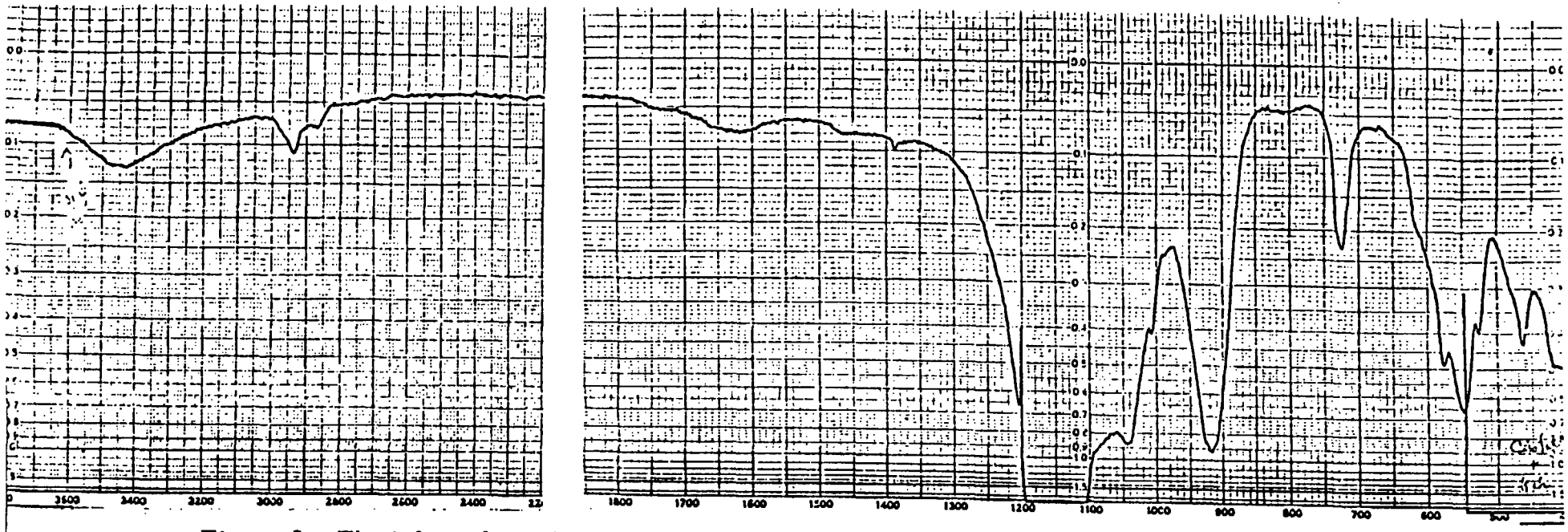

Figure 3. The infrared spectrum of the residue of an ashed Millipore® Celotate filter.

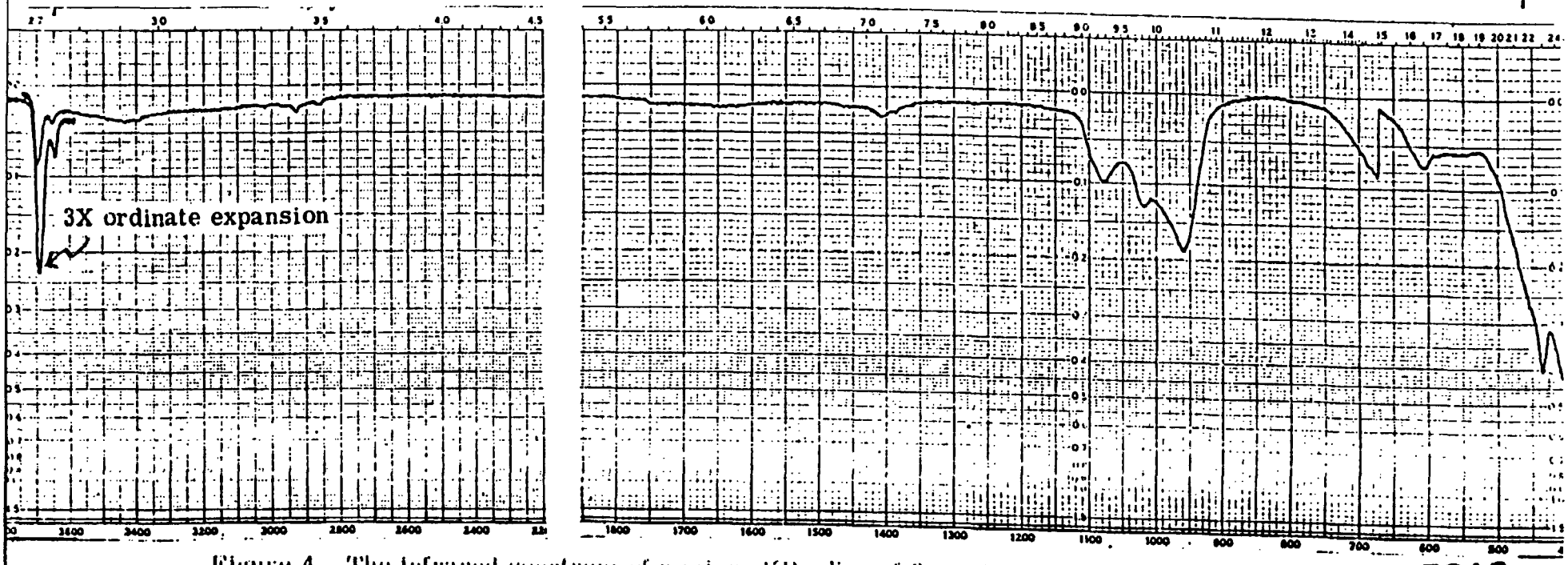

Figure 4. The infrared spectrum of a micro KBr disc of 3 µg chrysotile in 10 mg KBr.

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(Footnotes to Table I)

(a) Calculated from magnesium content using the formula $3MgO \cdot 2SiO_2 \cdot 2H_2O$ for chrysotile asbestos.

(b) Different sensitivities were obtained for the three filtered cell effluents because 500, 250 and 100 ml sample volumes were used for the different determinations.

It can be seen from Table I that samples having higher fiber counts also gave higher magnesium values. Also, the atomic absorption method appears to be a good tool for the determination of the efficiency of filtering systems for cell effluents. The time required for the determinations is of the order of 30 minutes per sample when batches of six or more are analyzed.

The sensitivity of the atomic absorption method is limited to 0.005 µg/ml of asbestos on a one liter sample due to the magnesium content of the Metricel* filter pad. The higher asbestos values obtained by atomic absorption are expected since one calculates all acid insoluble magnesium in the sample as chrysotile asbestos. It is known from the microscopy work that many particles present in cell effluent contain magnesium but are not chrysotile.

Silicon was determined in the above samples to see if a relationship existed between silicon content and asbestos.

Table II - Asbestos Levels in Cell Effluent As Calculated From Silicon Content

Unfiltered	Asbestos- µg/ml
10/23/74	21.2
10/24/74	90.7
10/29/74	32.5
Filtered	
10/22/74	<0.05
10/24/74	<0.05
10/29/74	<0.05

Table II demonstrates that there are other forms of silicon other than asbestos in the samples since lower asbestos levels are calculated from the magnesium data. This is not surprising since sand filters are used in the chlor-alkali process and the electrolysis cells are made of concrete. While a silicon determination could be used to evaluate the efficiency of a filtering operation, it is not as sensitive as the magnesium analysis and is more time consuming.

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In addition to the six samples of cell effluent, a sample of salt brine from the wells prior to the brine treatment plant was also analyzed and found to contain the equivalent of 0.023 µg/ml of asbestos. Referring back to Table I, it is seen that filtering the cell effluent will reduce the apparent asbestos content to less than that in the incoming brine.

Reference

H. A. Chagnard, Determination of Asbestos in Aqueous Samples by Atomic Absorption, LAD-AR-74.01.

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