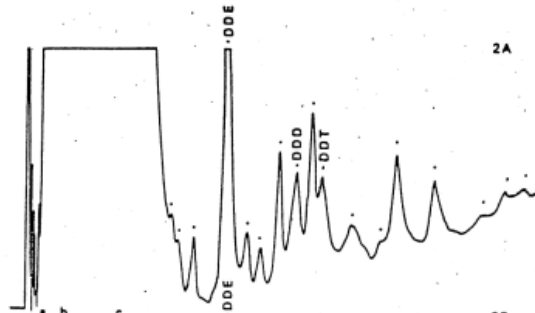


# CLEANUP FOR PCB ANALYSIS

- WHY?
- HOW?

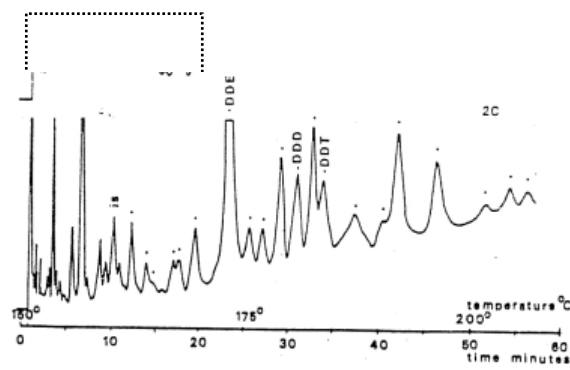
## BEFORE

Typical digested sewage sludge chromatogram showing severe sulfur contamination

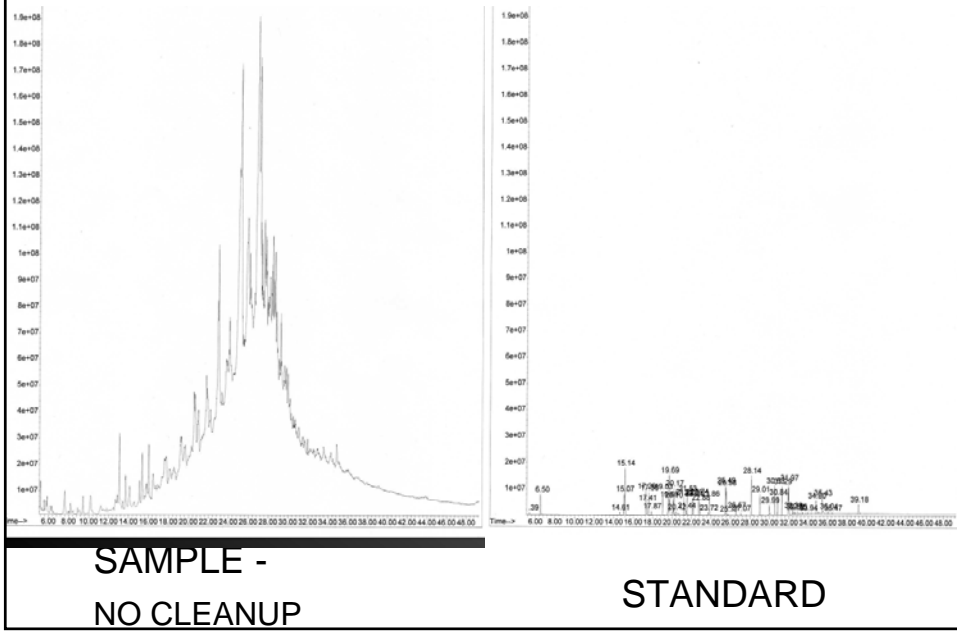


## AFTER

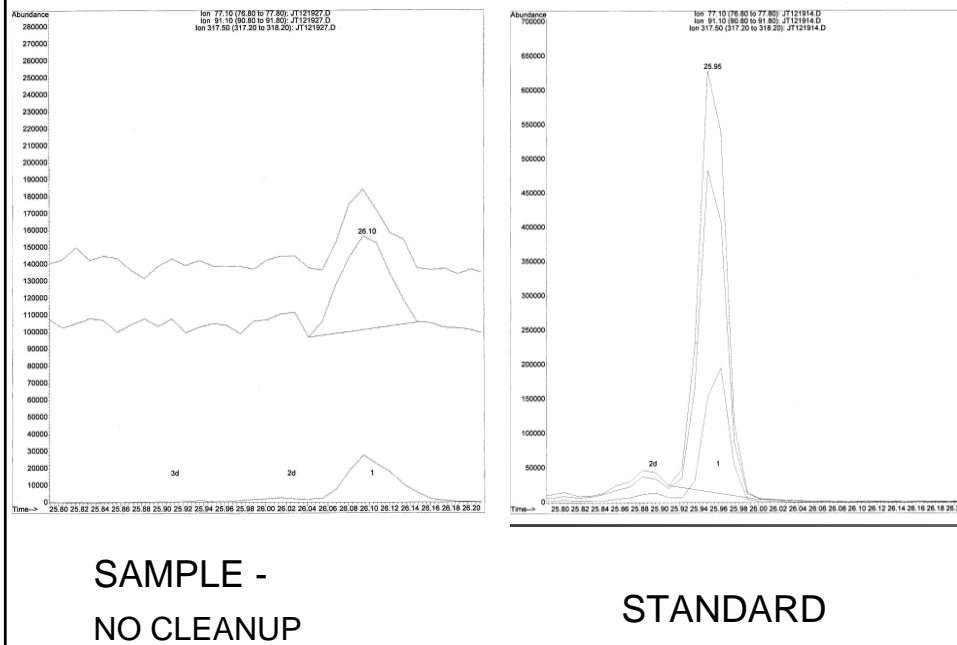
Final chromatogram after treatment with TBA-sulfite followed by sodium sulfite



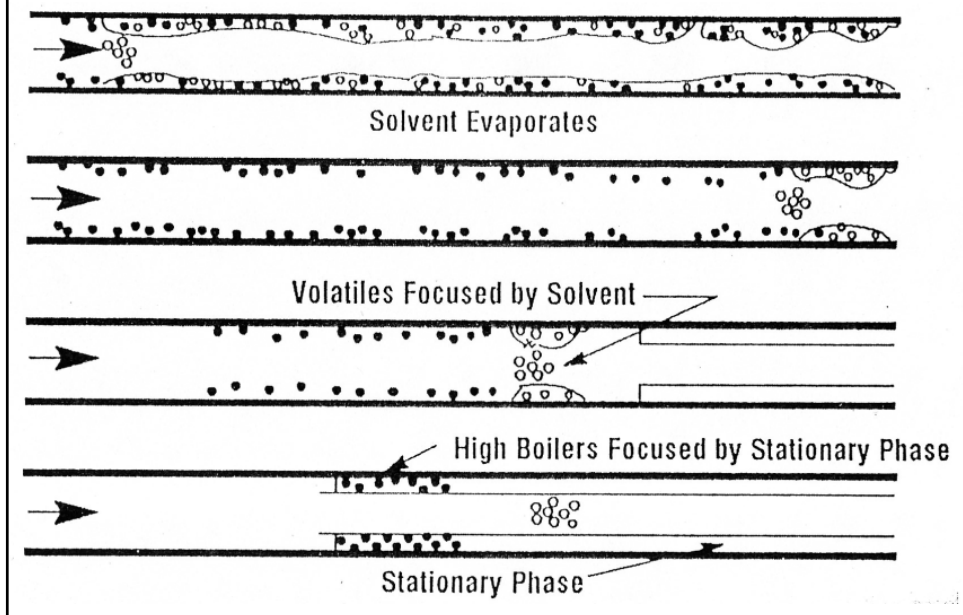
# TOTAL ION CHROMATOGRAMS



# SINGLE ION CHROMATOGRAMS



# FOCUSING AT THE INLET



**Figure 2-10**  
 Influence of a dirty vaporizing inlet (200°C). (Reproduced from K. Grob and G. Grob, *J HRC&CC*, 2 (1979) 109-117, with permission.)

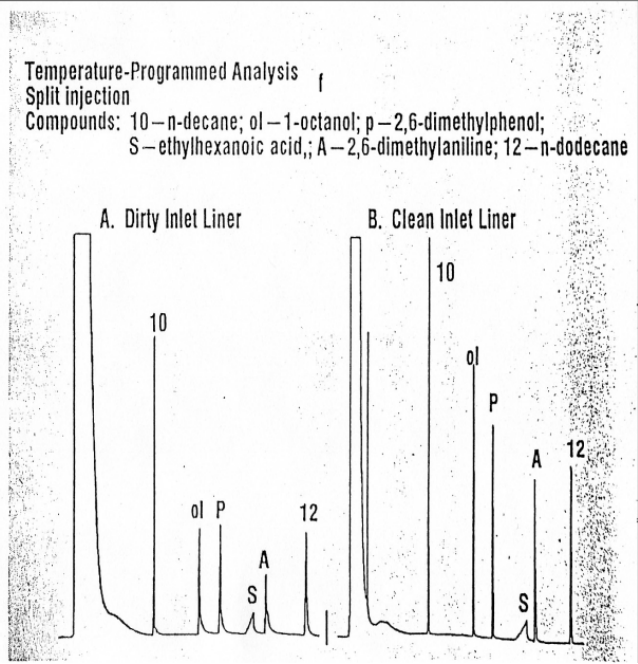
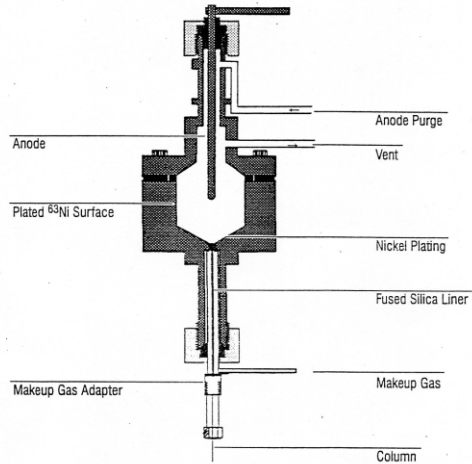


Figure 7-5

SIGNAL  
PROPORTIONAL  
TO DECREASE IN  
FLUX OF THERMAL  
ELECTRONS

IMPURITIES  
INCREASE FLUX  
OF THERMAL  
ELECTRONS =>  
PLUNGING  
BASELINE

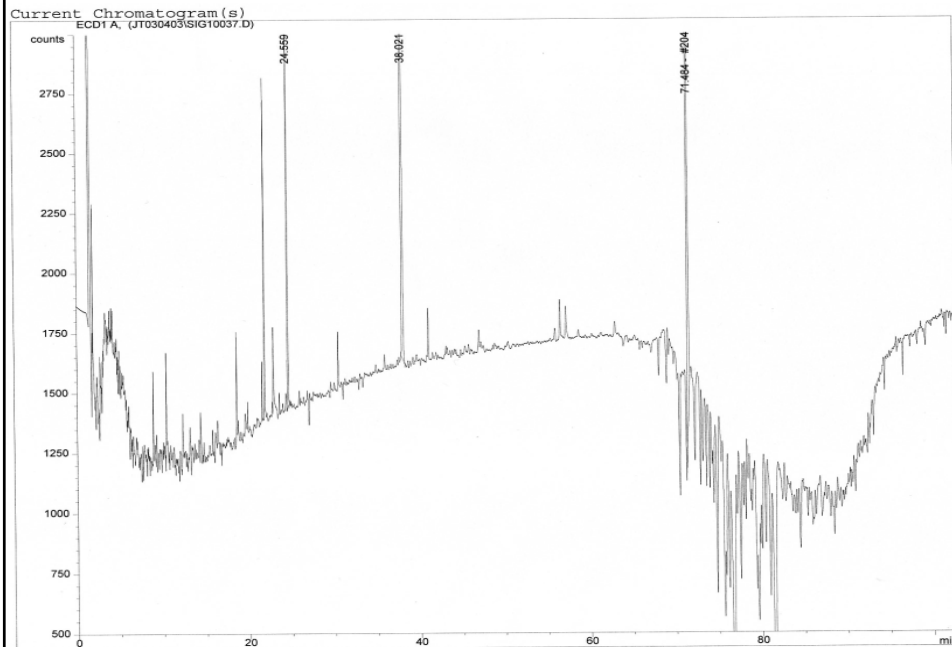


Electron Capture Detector (ECD)

The electron capture detector (ECD) cell contains  $^{63}\text{Ni}$ , a radioactive isotope emitting high-energy electrons ( $\beta$ -particles). These undergo repeated collisions with carrier gas molecules, producing about 100 secondary electrons for each initial  $\beta$ -particle.

Further collisions reduce energy of these electrons into the thermal range. These low energy electrons are then captured by suitable sample molecules, thus reducing total electron population within the cell.

## PLUNGING ECD BASELINE



# CLEANUP TECHNIQUES FOR PCBs

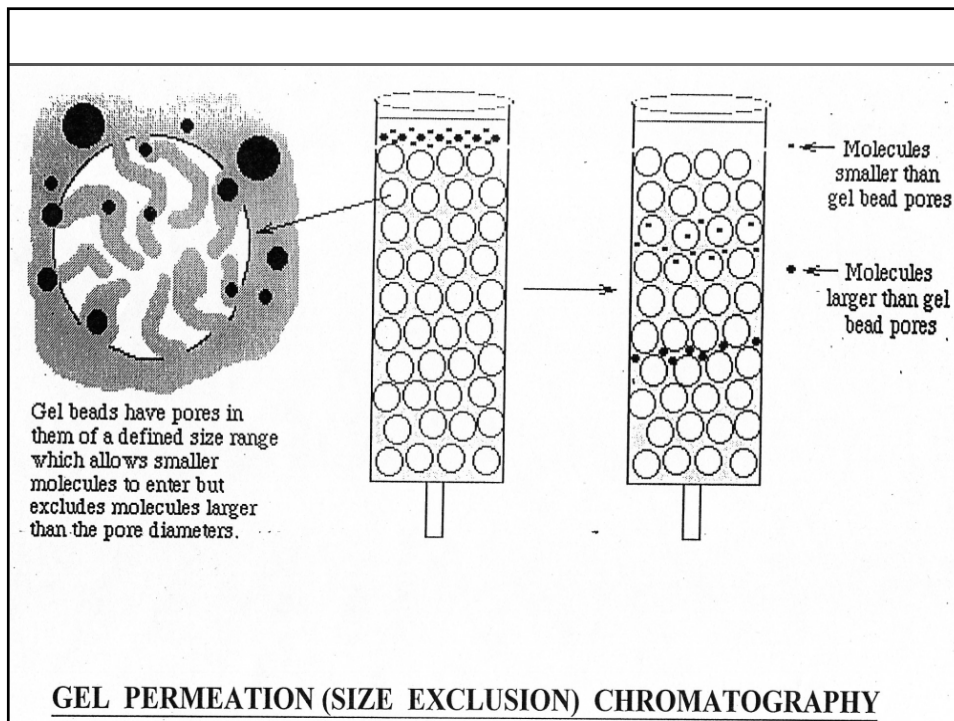
GEL PERMEATION CHROMATOGRAPHY

ADSORPTION CHROMATOGRAPHY

ALUMINA  
FLORISIL  
SILICA GEL

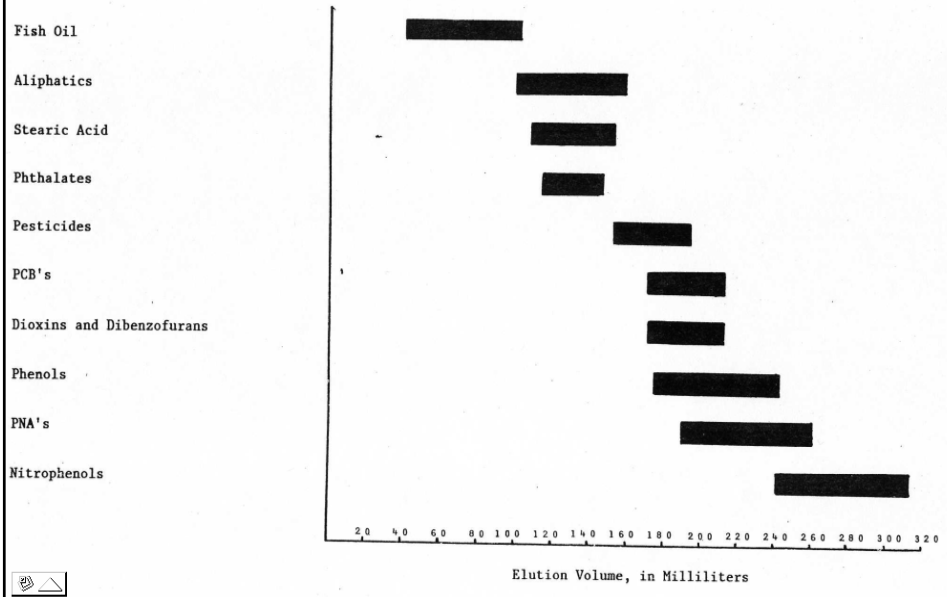
SULFURIC ACID

SULFUR REMOVAL



# GPC ELUTION PROFILE

Figure 1. Elution Volumes of Compounds from BioBeads SX-3, 60 grams, Eluted With Methylene Chloride:Cyclohexane (1:1).



## AN EXAMPLE OF GPC CLEANUP FOR PCBs

**PACKING: 60 g SX-3 Bio Beads 200-400 mesh**

**ELUTION SOLVENT :**  
**Cyclohexane / Methylene Chloride**

**1:1**

**FLOW : 5 mL / min**

**DUMP FOR : 24 minutes**

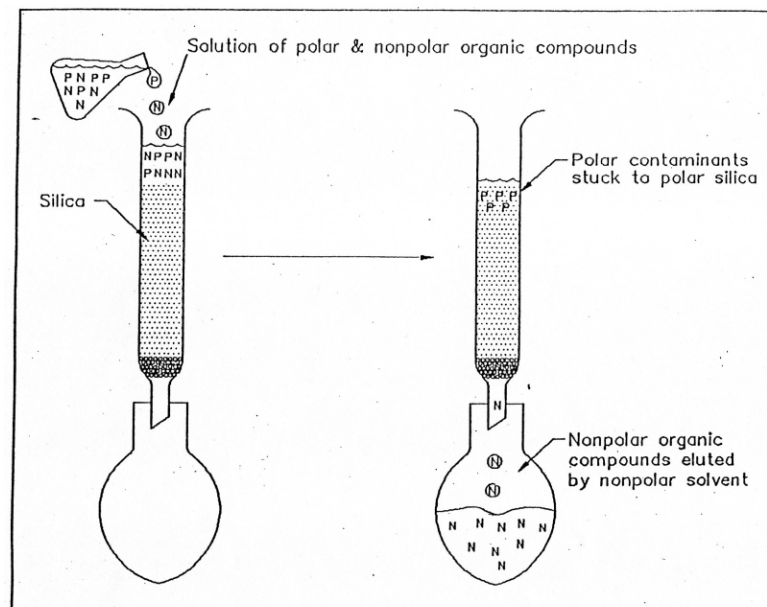
**COLLECT FOR : 40 minutes**

## **SOME ADSORBENTS FOR PCB CLEANUP**

**FLORISIL :  $\text{MgO} + \text{SiO}_2$**

**SILICA GEL :  $\text{SiO}_2 \cdot \text{H}_2\text{O}$**

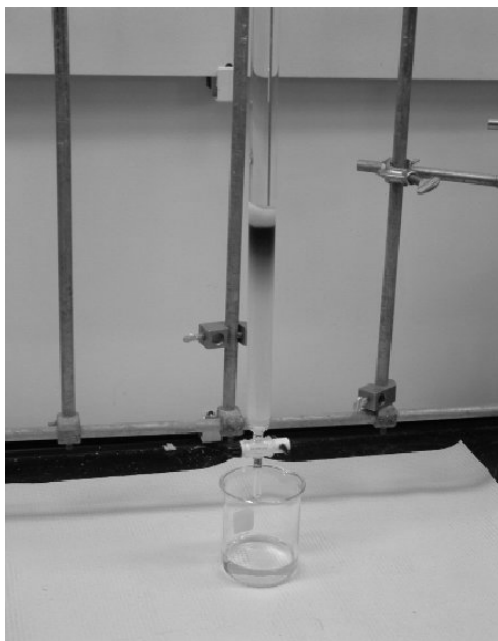
**ALUMINA :  $\text{Al}_2\text{O}_3$**



**ADSORPTION CHROMATOGRAPHY CLEANUP**

<u>ADSORBENT</u>	<u>ANALYTE</u>	<u>ELUTION SOLVENT</u>
FLORISIL	PCBs, DDT, ...	94% Hexane/ 6% Ethyl Ether
	PAHs	70% Hexane/ 30% Ethyl Ether
	Atrazine	50% Hexane/ 50% Ethyl Ether
SILICA GEL	PCBs	100% Hexane
	DDT	75% Hexane/ 25% Ethyl Ether
ALUMINA	Aliphatic Hydrocarbons	100% Hexane
	Aromatic Hydrocarbons	100% Methylene Chloride

## FLORISIL COLUMN CLEANUP





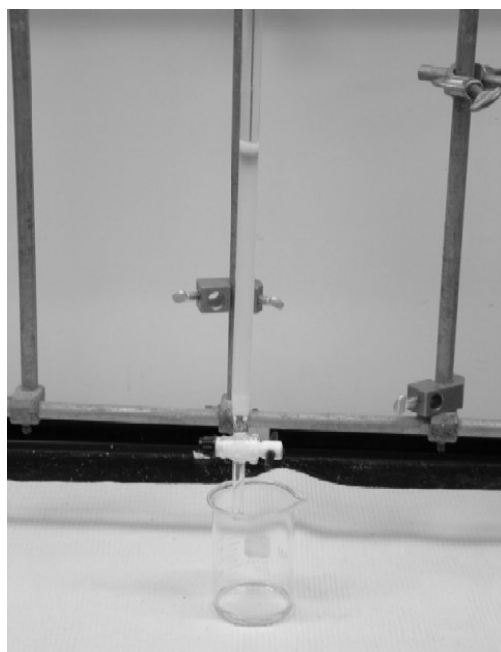
**AN EXAMPLE OF  
FLORISIL COLUMN CLEANUP  
FOR PCBs**

**PACKING: 22 g Florisil, 60-100 mesh,  
with a layer of anhydrous  
Sodium Sulfate on top**

**ELUTION SOLVENT : 94% Hexane /  
6% Ethyl Ether, 200 mL**

**ELUTION RATE : ~ 5 mL / min**

**SILICA GEL COLUMN CLEANUP**



**Silica Gel Deactivation Evaluation for Lot #016269.**

Fraction	Aroclor	Aroclor 1016/1260			
		3.3%	% Recovery	3.5%	% Recovery
SG-1	1016	1.76	88	1.77	88.5
	1260	2.32	98.3	2.31	97.9
SG-2	1016	Trace of 1016(<LOD)		Trace of 1016(<LOD)	
	1260	ND		ND	

Added 1.0mL of 1016/1260 at 2.00/2.36 mg/L to columns containing 5.0gms of deactivated silica gel at 3.3% and 3.5% respectively. The amount of aroclor in the SG-2 fraction indicates the need to continue deactivating at 3.5% for this lot of silica gel. The amount detected in the SG-2 fraction at 3.5% deactivation was negligible.

Fraction	Pesticide	Pesticides			
		3.3%	% Recovery	3.5%	% Recovery
SG-1	pp'DDE	0.118	102	0.123	106
	pp'DDT	ND		ND	
SG-2	trans-Chlordane	0.097	88.2	0.097	88.2
	Dieldrin	0.093	93	0.098	98

Added 3.0mL of "Level 5 Pesticide Std." to columns containing 5.0gms of silica gel deactivated at 3.3% and 3.5% respectively. Results indicate that deactivating at 3.5% effectively puts most of the analyzed pesticides in the appropriate fraction. trans-Nonachlor should split between the two fractions at roughly 15/85%.

**AN EXAMPLE OF  
SILICA GEL COLUMN CLEANUP  
FOR PCBs**

**PACKING: 5.0 g Silica gel, 100-200 mesh,  
deactivated with 3.5% water  
by weight,  
with a layer of anhydrous  
Sodium Sulfate on top**

**ELUTION SOLVENT : 100% Hexane,  
50 mL**

**ELUTION RATE : ~ 5 mL / min**

**AN EXAMPLE OF  
ALUMINA COLUMN CLEANUP  
FOR PCBs**

**PACKING: 10.0 g Alumina, neutral,  
“Brockman Activity I” ,  
60 – 325 mesh,  
with a layer of anhydrous  
Sodium Sulfate on top**

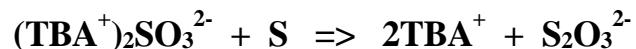
**ELUTION SOLVENT for ALIPHATICS :  
100% Hexane, 15 mL**

**ELUTION SOLVENT for PCBs : 100%  
Methylene Chloride, 100 mL**

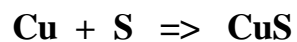
**ELUTION RATE : ~ 2 mL / min**

**SOME TECHNIQUES FOR THE REMOVAL OF  
ELEMENTAL SULFUR FROM PCB EXTRACTS**

**Tetrabutylammonium sulfite (TBA) ;**



**Copper ;**



**Mercury;**

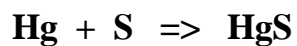


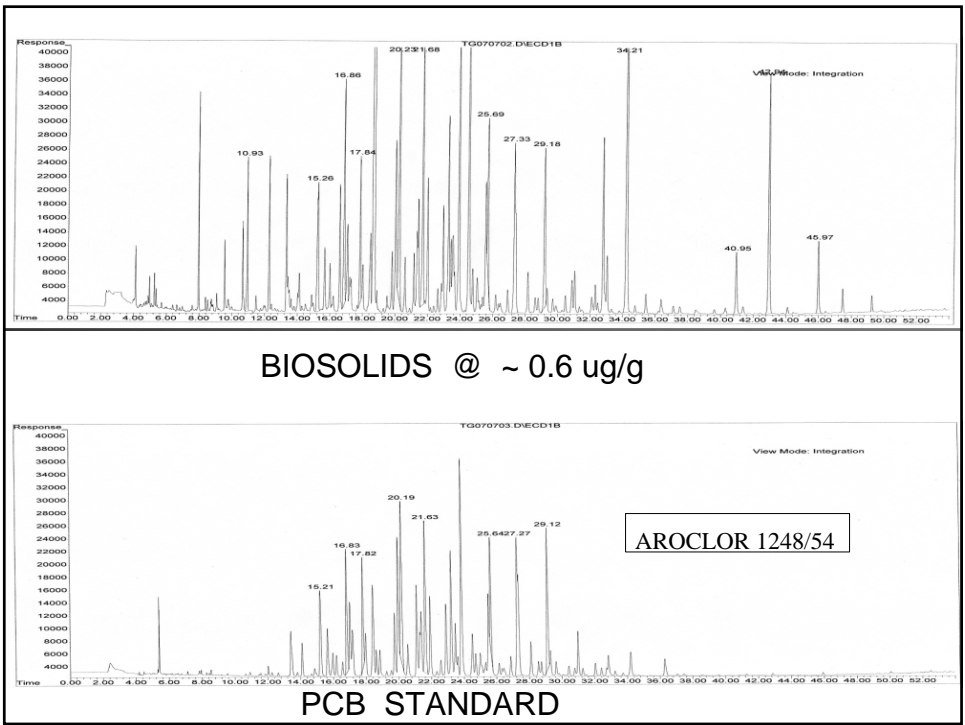
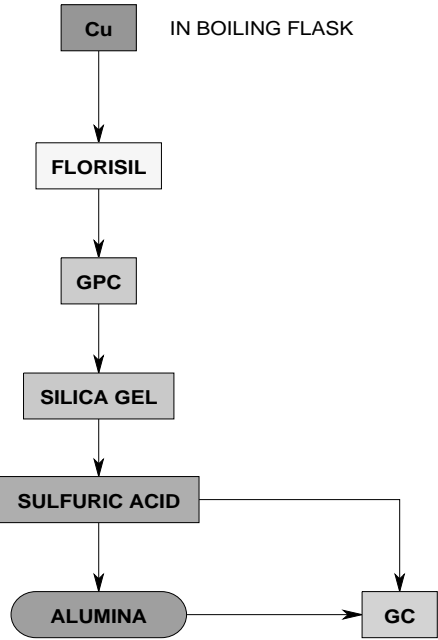
Table 1. EFFECT OF MERCURY AND COPPER ON PESTICIDES

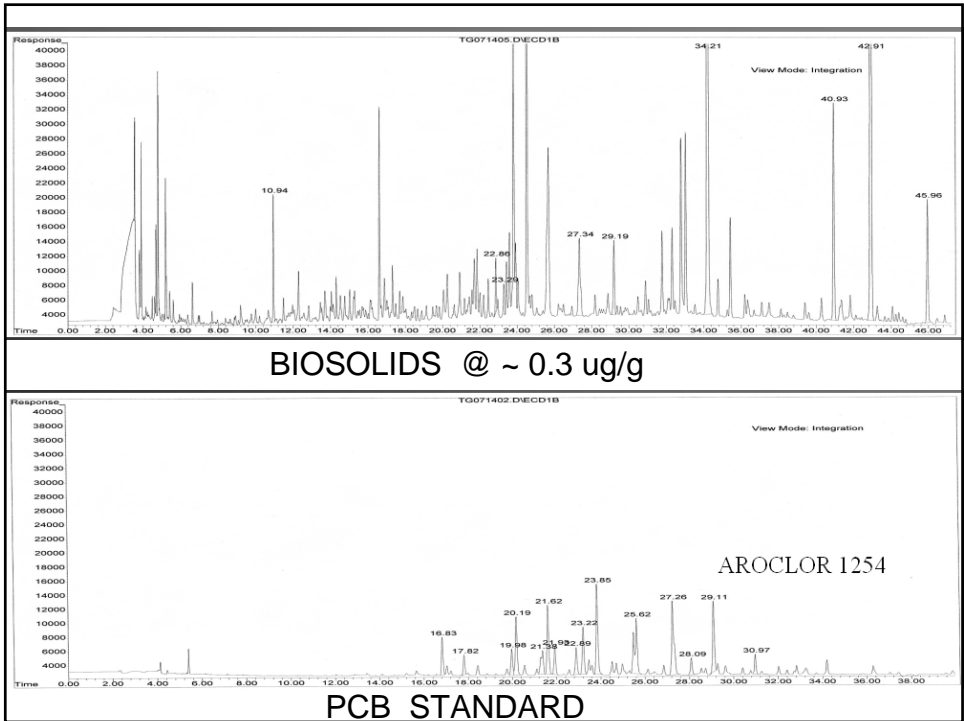
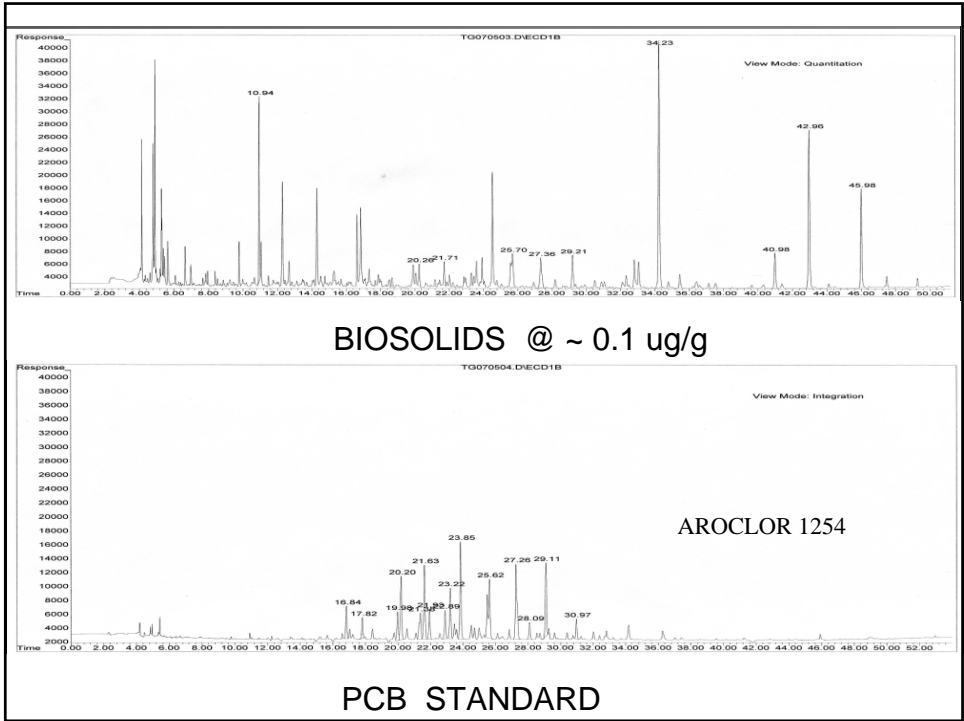
Pesticide	Percent Recovery <sup>a</sup> using:	
	Mercury	Copper
Aroclor 1254	97.10	104.26
Lindane	75.73	94.83
Heptachlor	39.84	5.39
Aldrin	95.52	93.29
Heptachlor epoxide	69.13	96.55
DDE	92.07	102.91
DDT	78.78	85.10
BHC	81.22	98.08
Dieldrin	79.11	94.90
Endrin	70.83	89.26
Chlorobenzilate	7.14	0.00
Malathion	0.00	0.00
Diazinon	0.00	0.00
Parathion	0.00	0.00
Ethion	0.00	0.00
Trithion	0.00	0.00

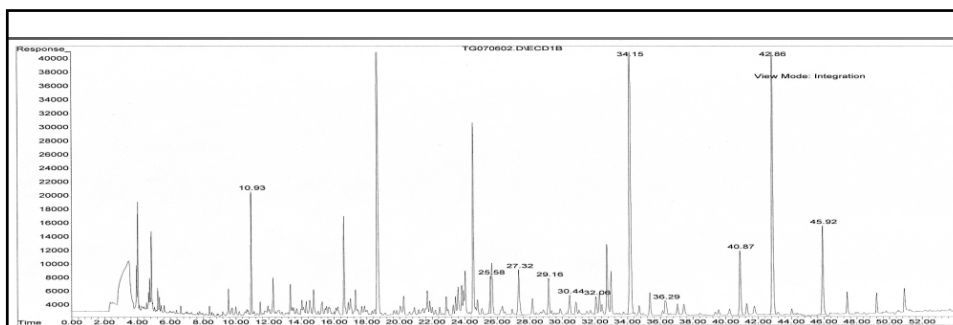
<sup>a</sup> Percent recoveries cited are averages based on duplicate analyses for all compounds other than for Aldrin and BHC. For Aldrin, four and three determinations were averaged to obtain the result for mercury and copper, respectively. Recovery of BHC using copper is based on one analysis.



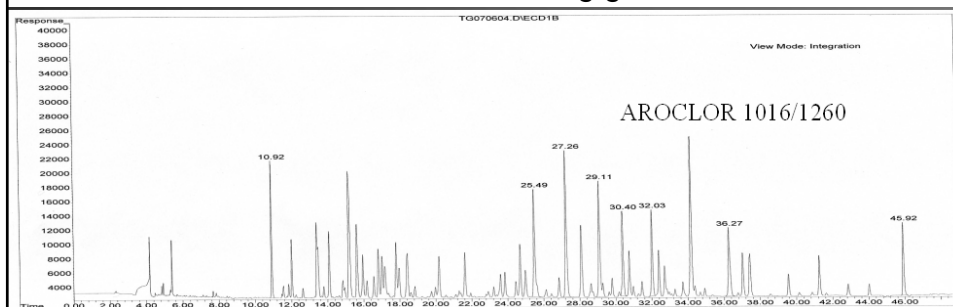
# TYPICAL CLEANUP SCHEME FOR BIOSOLIDS



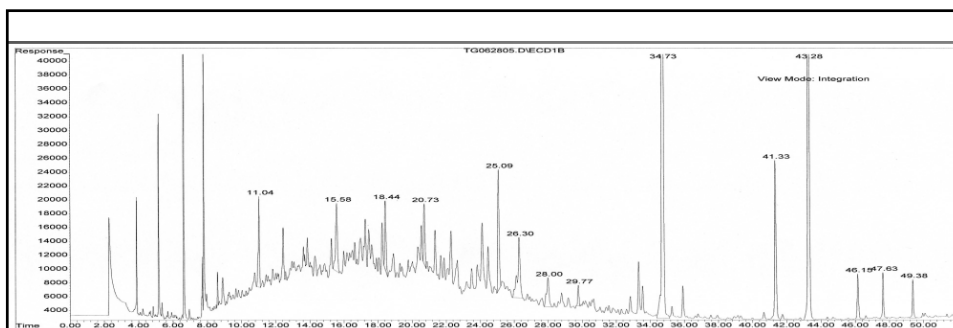




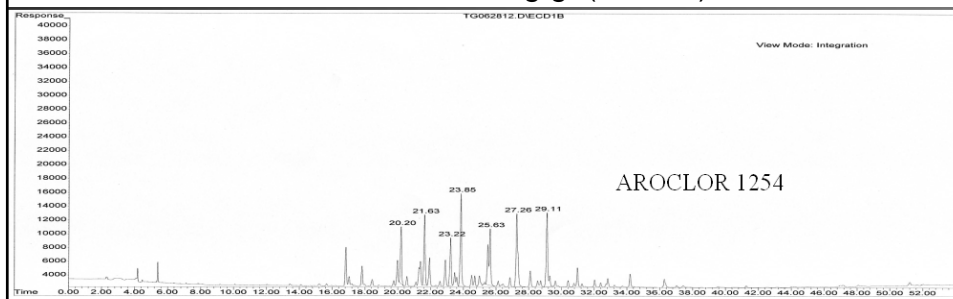
BIOSOLIDS @ ~ 0.1 ug/g



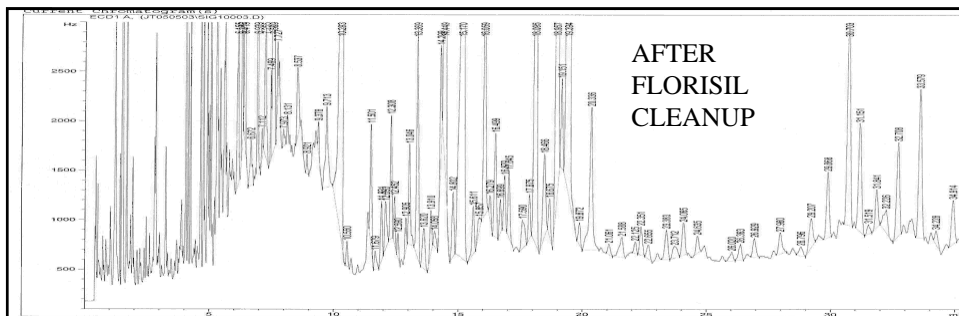
PCB STANDARD



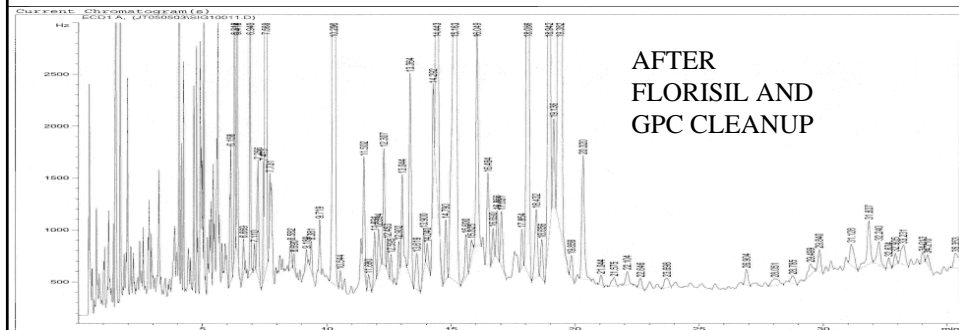
BIOSOLIDS @ ? ug/g (\*I < 0.2)



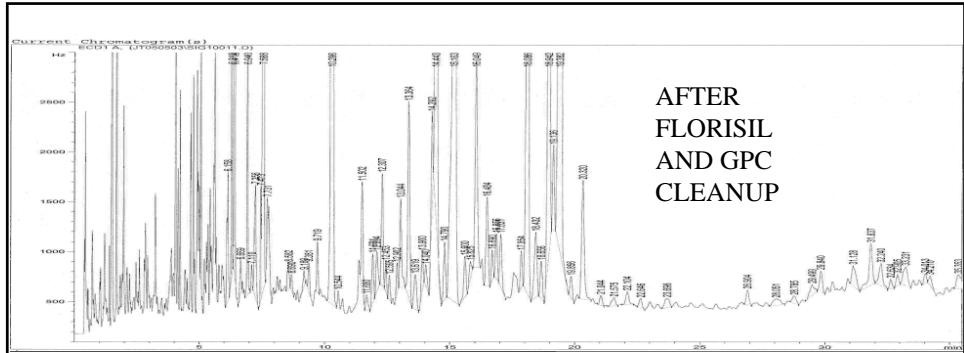
PCB STANDARD



PAPER MILL SLUDGE EXTRACT

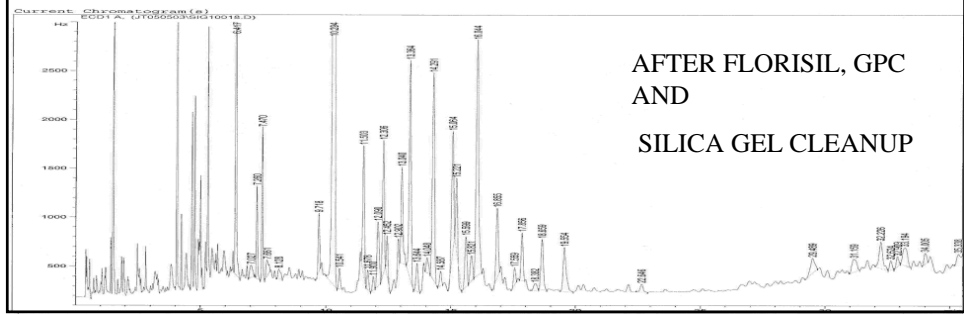




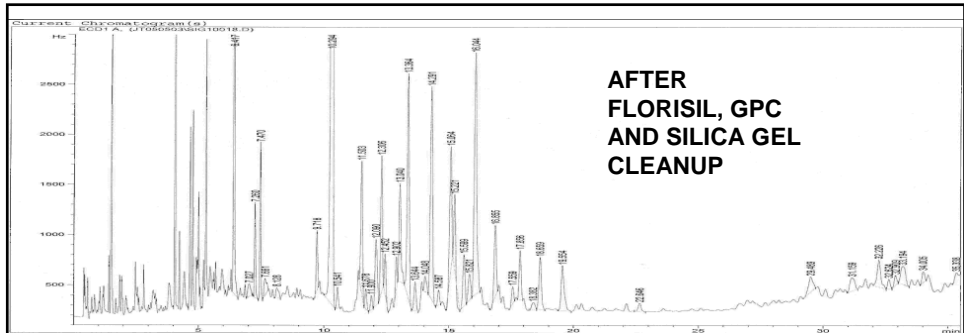


AFTER  
FLORISIL  
AND GPC  
CLEANUP

**PAPER MILL SLUDGE EXTRACT**

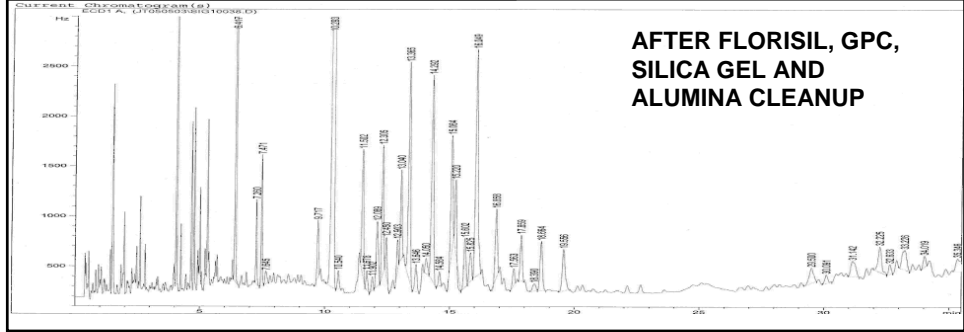


AFTER FLORISIL, GPC  
AND  
SILICA GEL CLEANUP

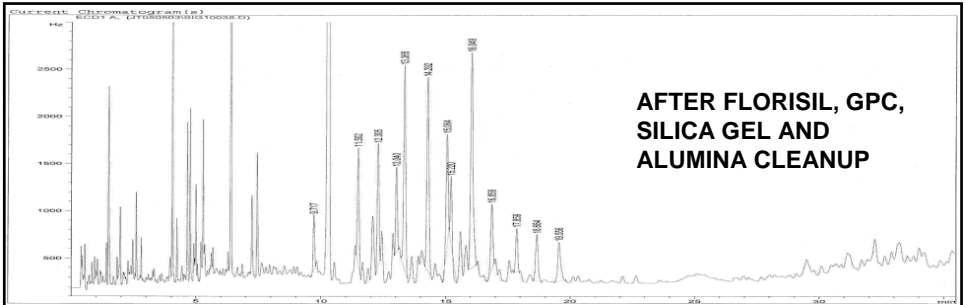


AFTER  
FLORISIL, GPC  
AND SILICA GEL  
CLEANUP

**PAPER MILL SLUDGE EXTRACT**

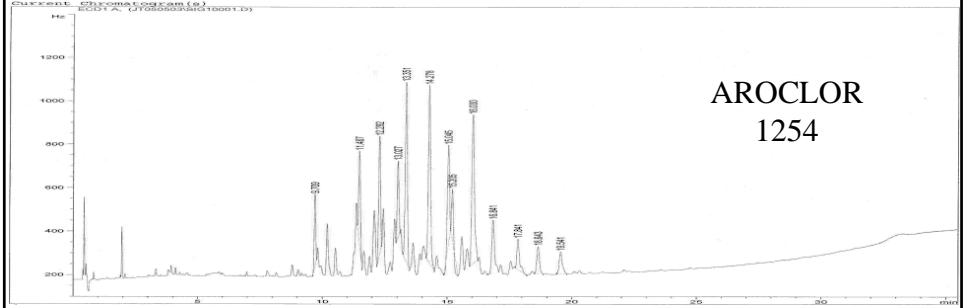


AFTER FLORISIL, GPC,  
SILICA GEL AND  
ALUMINA CLEANUP



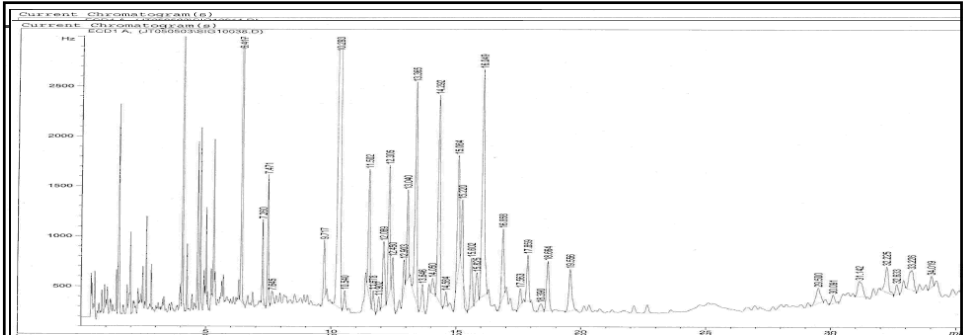
**AFTER FLORISIL, GPC,  
SILICA GEL AND  
ALUMINA CLEANUP**

**PAPER MILL SLUDGE @ ~ 0.1 ug/g**

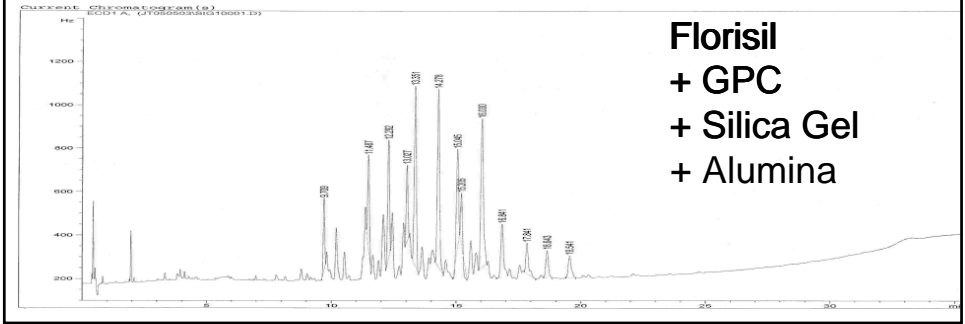


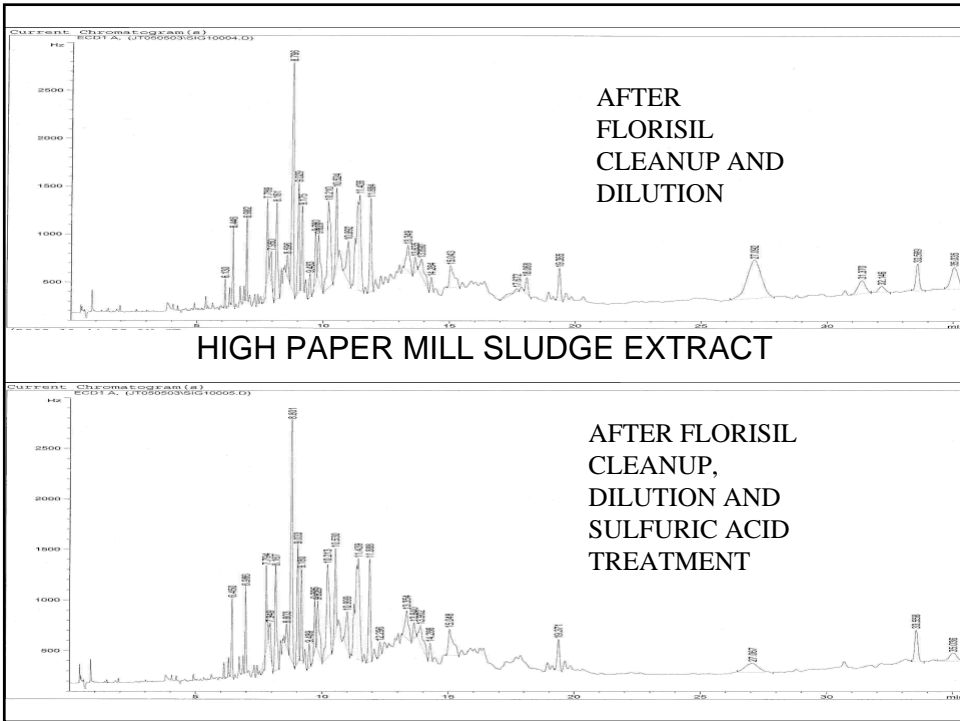
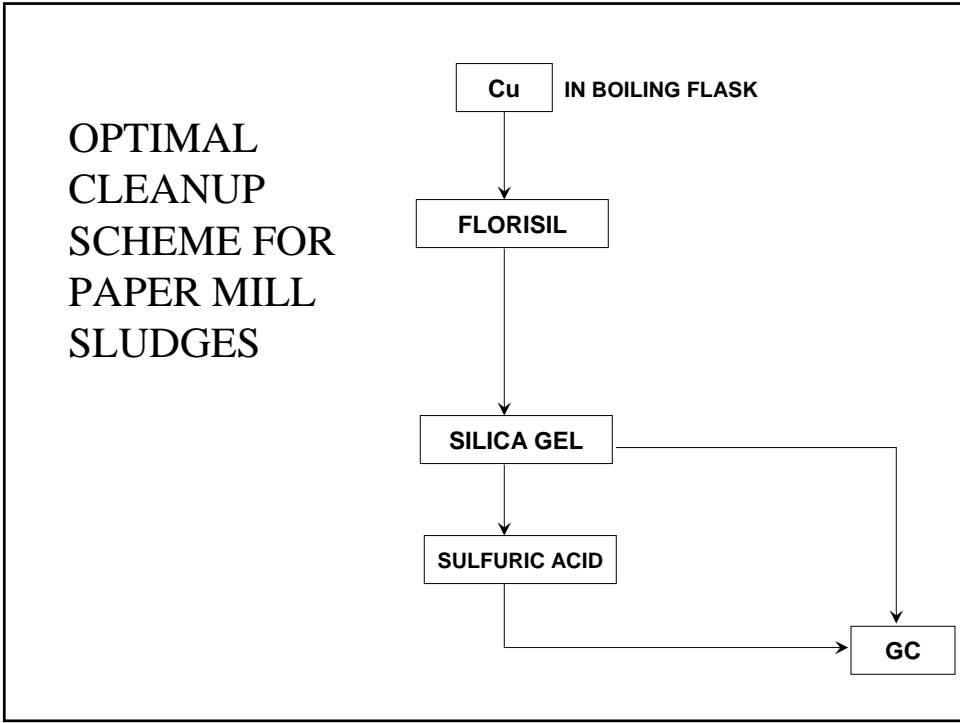
**AROCLOR  
1254**

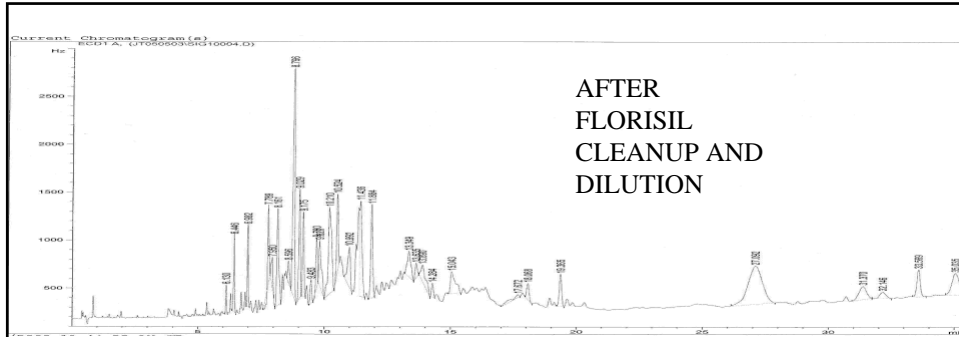
**PCB STANDARD**



**Florisil  
+ GPC  
+ Silica Gel  
+ Alumina**

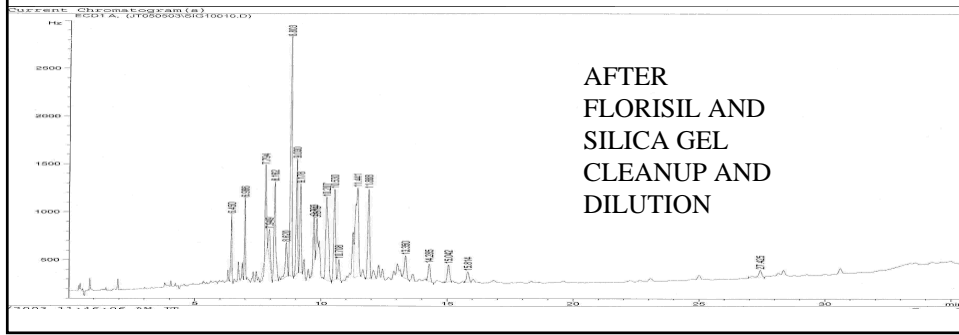




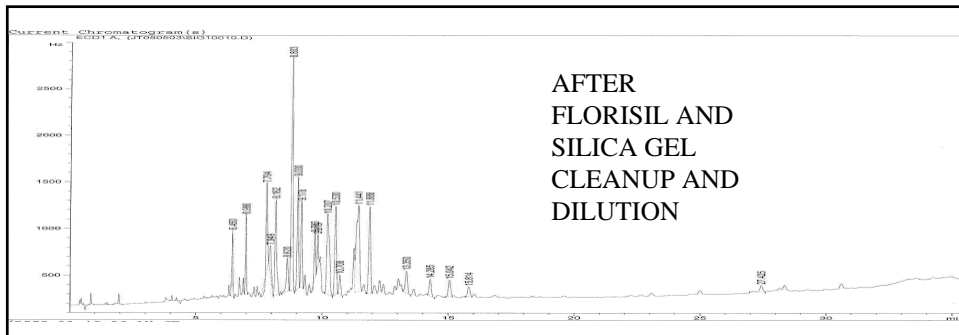


AFTER  
FLORISIL  
CLEANUP AND  
DILUTION

HIGH PAPER MILL SLUDGE EXTRACT

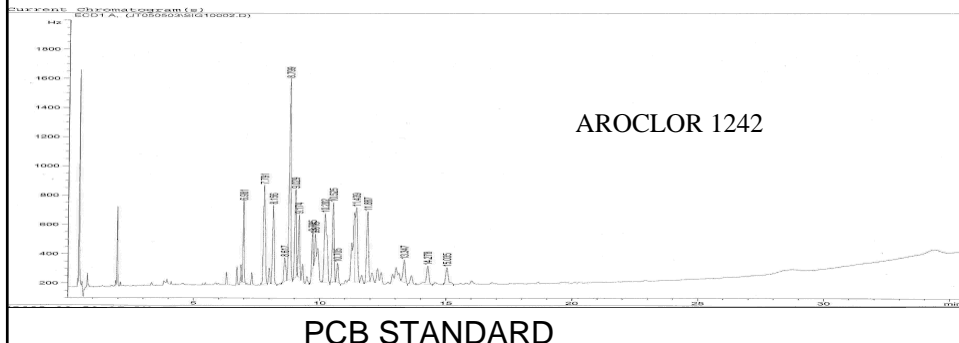


AFTER  
FLORISIL AND  
SILICA GEL  
CLEANUP AND  
DILUTION



AFTER  
FLORISIL AND  
SILICA GEL  
CLEANUP AND  
DILUTION

PAPER MILL SLUDGE @ ~ 5 ug/g



AROCLOR 1242

PCB STANDARD

