US EPA RECORDS CENTER REGION 5
515296

Life Systems, Inc.

24755 HIGHPOINT ROAD · CLEVELAND, OHIO 44122 · PHONE: 216 464-3291

August 12, 1982
RHR-8-10

Dr. Timothy G. Shea
Director of Environmental Engineering
Engineering-Science Company
7903 Westpark Drive
McLean, VA 22102

Subject: Sampling and Analytical Protocol Notes

References: 1. ICAIR Technical Directive No.: 05
2. Meeting between Mr. Mike Kosakowski of EPA and Mr. Bob Rosing of LSI on 08/03/82

Dear Dr. Shea:

Enclosed is a copy of the Sampling and Analytical Protocol Notes, D-117 prepared by Dr. Brenda Kimble. These are rough notes from Dr. Kimble's two-day protocol development meeting with Capsule Laboratories in Minneapolis, MN on August 26-27, 1982. It was agreed upon during the above referenced meeting that these notes would be submitted as received by LSI. This protocol will be finalized under a future agreement.

If you have any questions, please contact me by calling (216) 464-3291.

Very truly yours,

LIFE SYSTEMS, INC.

R. H. Reuter, Ph.D.
Vice President

RHR/cdr

encl.: Sampling and Analytical Protocol Notes, D-117

cc: ✓Ms. Julie Klaas, w/encls. (5 copies)
Procurement File
Task Manager (TD 051238)

D-117

SAMPLING AND ANALYTICAL PROTOCOL NOTES

Technical Directive No.: 05
Technical Directive Title: Reilly Tar Technical Support

Technical Directive Report

by

Dr. Brenda Kimble

for

ICAIR SYSTEMS DIVISION
LIFE SYSTEMS, INC.
24755 Highpoint Road
Cleveland, OH 44122

August 1, 1982

STUDY PROTOCOL

I Introduction

Executive Summary

rationale

why the protocol is the way it is

III Field Sampling

- grab samples for volatiles
- resin bed samples
- polluted samples

Δ cost

IV Analytical Chemistry

(per sample cost)

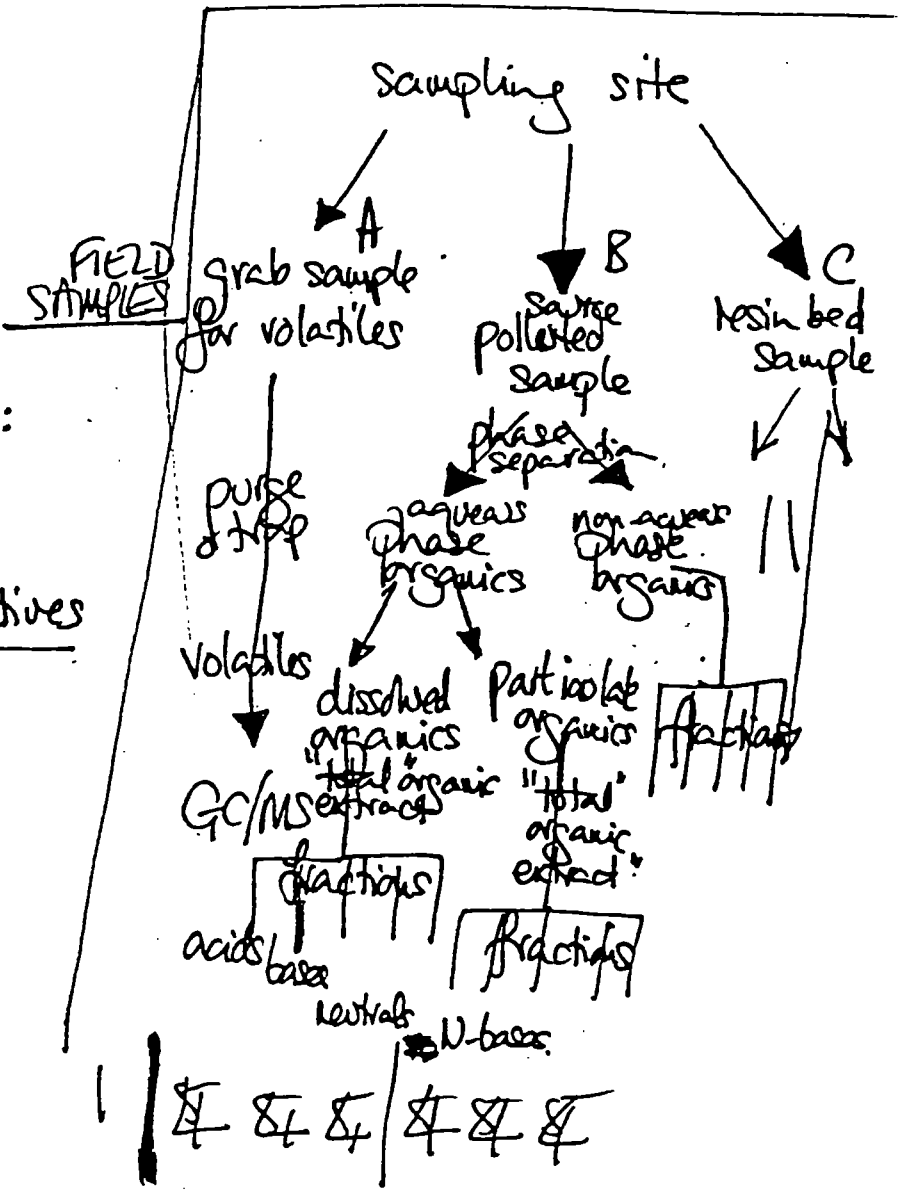
- ~~phase separation~~ extraction
- fractionation to acids/bases/neutrals
- volatiles → fractionation of bases
- GC/MS
- data processing

V QA/QC

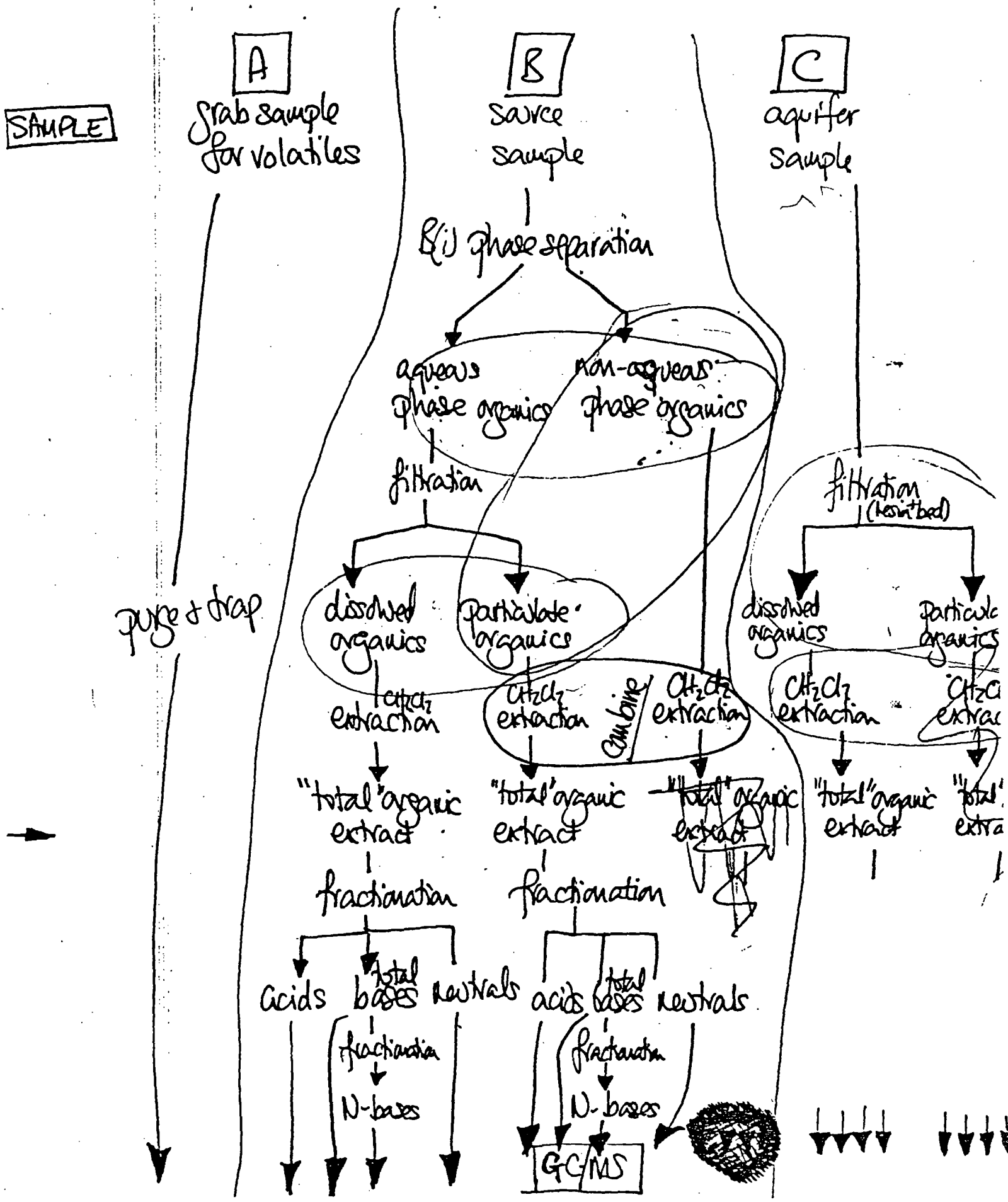
VI Chain of Custody

VII Information Required:

Appendix: Study Alternatives



SUMMARY OF ANALYTICAL PROTOCOL



IV Analytical Chemistry

A. Volatiles: (1624)

purge and trap
cap. GC/MS
instrument parameters

int. std: d_6-benzene, p-fluorobromobenzene
deuterated target compound ?
methylnaphthalene ?

B. Source Sample(s)

(i) phase separation (gravity) 24 hrs. burette-column, semi-quantitative measure volume ratios drain slowly
~~filtration~~ ?
(ii) CH_2Cl_2 + ether extraction of ~~dissolved and particulate organics~~ aqueous phase
~~" " " particulate organics~~
(iii) CH_2Cl_2 + ether " " non-aqueous phase organics
(iv) fractionation into acids / bases / neutrals
(v) GC/MS analysis of total bases
(vi) isolation of nitrogenous bases
(vii) GC/MS : cap, instrument parameters
(viii) data processing

int std d_{10}-anthracene
deuterated target compounds: acid
N-base
S-compd
PAH

SUMMARY

GC/MS

Grab samples for volatiles:
Std spiked mixture for volatiles ← MDL, MS validation — 1 per st

Source sample : aqueous phase organics
- A
- total B
- N
- N-bases

non-aqueous phase organics
- A
- total B
- N
- N-bases

} 8

Aquifer samples :
- A
- total B
- N
- N-bases

} 4

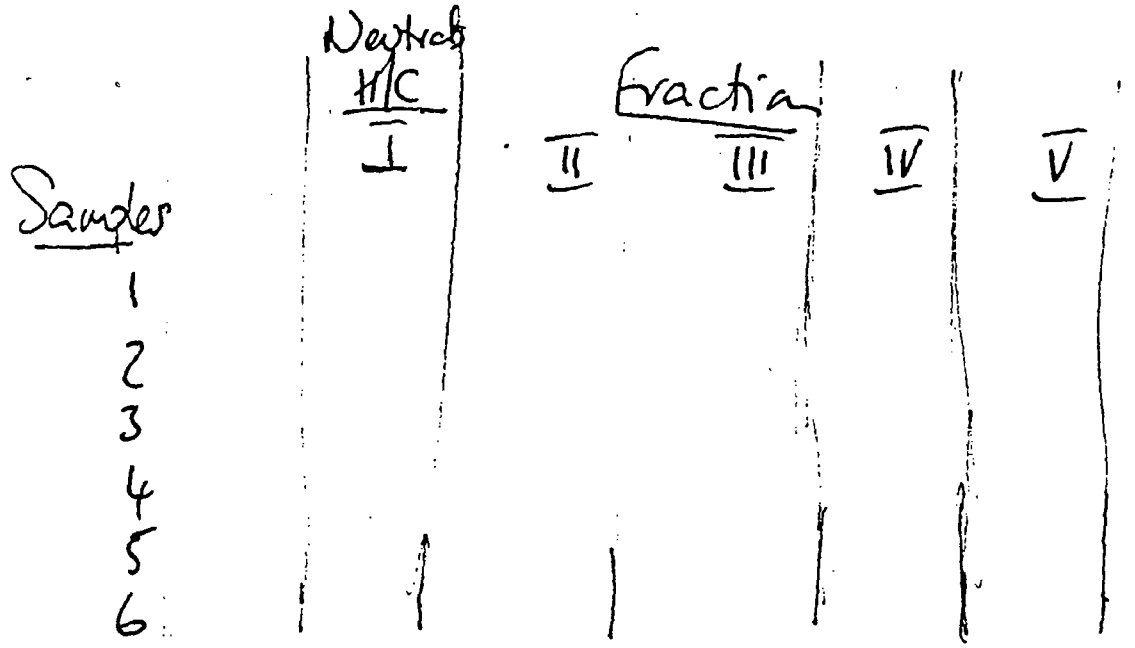
Sampling control :
(clean well)
- A
- total B
- N
- N-bases

} 4

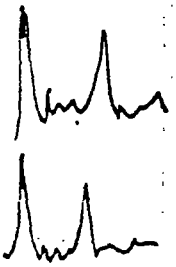
Standard mixture (from toxicol. list)
(fractionation method blank)
(spiked)
- A
- total B
- N
- N-bases

} 4

Unfractionated std mixture dilutions (spiked) for MDL etc. — n



S profiles



- Neutrals
- Bases
- Acids
- (Volatiles)

1 hexane
2 benzene
3 CH_2Cl_2
4 $CH_2Cl_2 : CH_3OH$ 1:1
5 CH_3OH

} Dissolved / Particulates

N- caupands
S- caupands (USGS S-caupand work ?)

Requirements for pumping sites
Drinking water wells
Copy of USGS Report → Capsule Labs ?

Publishing results ?

Weather during samples

Sampling by End of October

[redacted] Dec Jan Feb — no sampling
Nov March — ?

City supply wells — OK (in buildings)

Options:

✓ eliminate volatiles
combine aqueous + non-aqueous phase organics (homogenize
✓ combine dissolved + particulate organics
eliminate N-base fractionation

Deuterated Standards (from EPA?)

Volatiles	Acids	Bases	Neutrals
	p-cresol	acridine	benzo(a)pyrene
		~~quinoline~~	benzo-b-thiophene

Internal Standard

d_{10}- anthracene

VII Information Required

Deuterated standards from EPA
List of key compounds: (a) wood chemists to choose
(b) toxicologists
Sample sites (whether 2-phase sample?)
previous data on quantitation

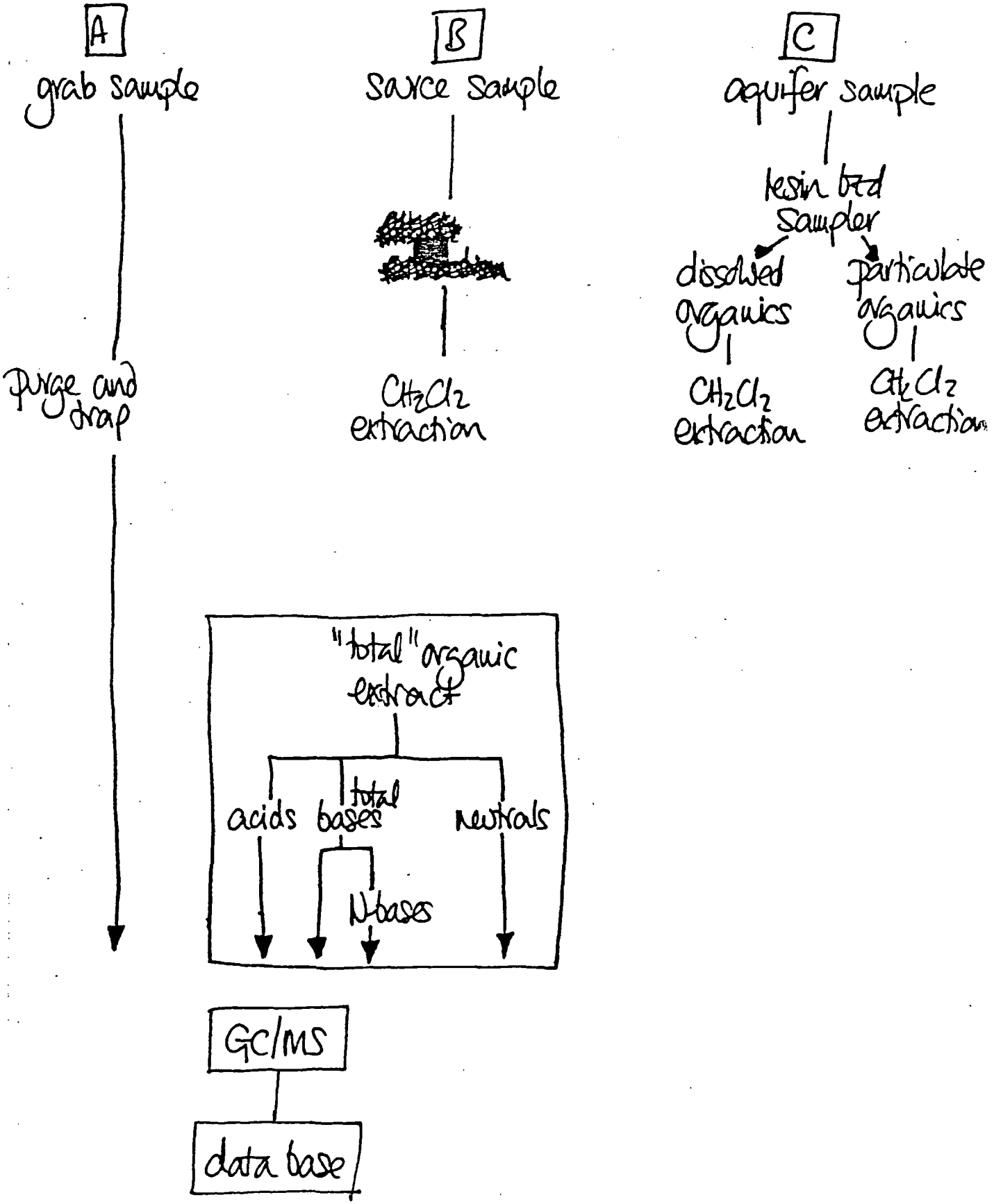
Permit / surcharge to run wells to discharge to
sanitary sewer — contact City of SLP

Weather (before end of October)

Well 5 — no where to discharge water

Sufficient diameter for observation wells
without submersible pump

SUMMARY OF ANALYTICAL PROTOCOL



Per site

		GC/MS
		VOAS
Grab sample for volatiles		1
Source samples	min (total CH_2Cl_2 extract)	4 (acids, total bases, N-bases, neutral)
	+ (dissolved + part)	4x2
	+ (aq. + non-aq)	4x3
		4 – 12
aquifer samples		4 – 8

4 – 13 per site

1	13	9
2	9	5
3	9	5
4	9	5
5	9	5
6	9	5
	58	34

58 + standards etc.

C. <u>Aquifer Samples</u>

(i) MeOH elution of resin bed
(ii) CH_2Cl_2 elution of resin bed
(iii) combine eluates and dry with Na_2SO_4 to obtain CH_2Cl_2 extract of resin bed
(iv) Soxhlet extraction of filter with CH_2Cl_2 to obtain CH_2Cl_2 extract of particulates
(v) combine CH_2Cl_2 extracts of particulates and resin bed

0.125 amu
35 μsec
check with Chris

Biller/Biemann
plot flagged spectra
new system acquisition rates

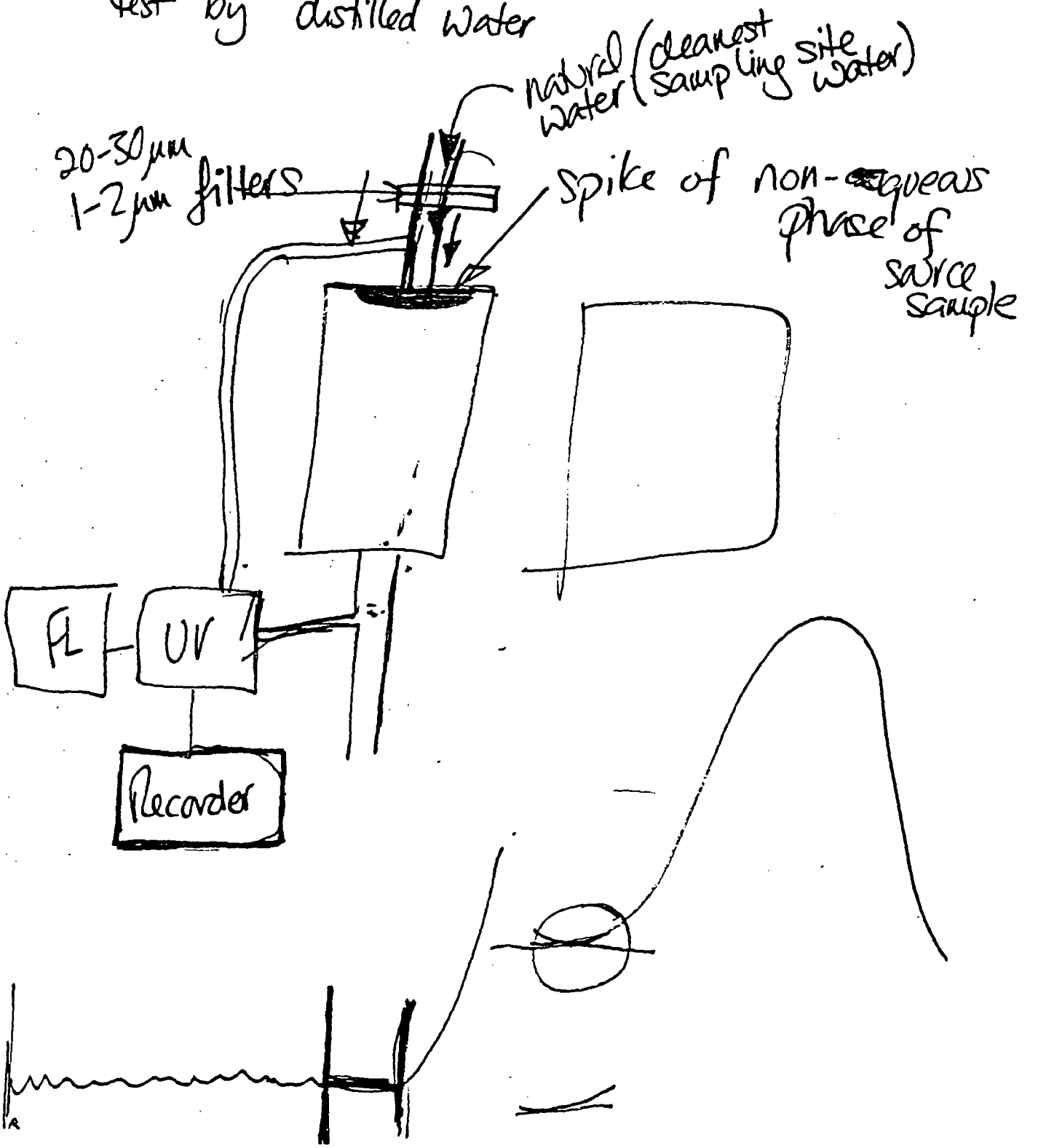
M. Godsey (mass spec)
USGS, Menlo Park

Sampling

either : 8 resin beds off a manifold

or : larger resin beds (not validated)

test by distilled water



Unfractionated std mixture (spiked) spectrometer validation

M

Assume 6 sites			DP		Hours/run
					volatile stds
6 volatiles	6	+	stds 6		6×2 = 12
14 1 source sample	8		8		8×2 = 16
28 5 aquifer samples	20		20	72 hrs	20×2 = 40
42 1 sampling control	4		[redacted]		4×2 = 8
fractionated std mixture	4		1		4×2 = 8
	42		2		
MDL	16				16×1 = 16
MS validation	14		2		14×1 = 14
	72		40 hrs.		

(2 per day; 50% of sample time)

72 in 3 weeks (34)

5 runs/day for 14.5 days

114 hrs = 14.25 days.

say 20×8 = 160 hrs

GC/MS running time
data processing

		hr	
Da	std	1	
	blk		½
	sample 1	2	
	sample 2	2	
	blank		½
	sample 3	2	
	sample 4	2	
	std	1	

11[redacted] hrs x 9 days = 99 hrs

MDL 16 hrs 16 hrs

116 hrs.

system blank
std
3 samp 1
4 blk
5 samp 2
6 blk
7 samp 3
8 blk
9 std

Volatiles:

6 samples x 2 hrs
2 stds
6 blanks x 2 hrs

18 hrs / 3 samples 36 hrs.

152 hrs