IA COUNTY
H CARE SERVICES
AGENCY
DAVID J. KEARS, Agency Director

ENVIRONMENTAL HEALTH SERVICES
ENVIRONMENTAL PROTECTION
1131 Harbor Bay Parkway, Suite 250
Alameda, CA 94502-6577
(510) 567-6700
FAX (510) 337-9335

SR 0003775

July 17, 2003

Douglas L. Kasefang
Senior Environmental Project Manager
Albertsons Inc.
P.O. Box 20
Boise, Idaho 83726

RE: Closure of one underground storage tank, Dublin Data Center, 6300 Clark Avenue, Dublin, California 94568

Dear Mr. Kasefang:

Thank you for the Tank Closure Report dated May 30, 2003, prepared and submitted by SECOR International, Inc. for the above referenced site.

The report provides the results of the activities conducted during the removal of a 2,000-gallon underground storage tank. Following the review of this report, this department is satisfied that the former tank has been closed in full compliance with the requirements of Title 23, California Code of Regulations.

The referenced report documents that no apparent and/or significant release from the former tank has occurred at the site. Therefore, no further investigation or cleanup actions are required. Please be aware that further work may be required if conditions change or a water quality threat related to the tank is discovered at the subject site.

If you have any further questions concerning this matter, please contact me at (510) 567-6781.

Sincerely,

Robert Weston
Sr. Hazardous Materials Specialist

c: Susan Hugo, Manager, ACDEH
Neil Doran, SECOR International, Inc.

AC-19

STATE OF CALIFORNIA
STATE WATER RESOURCES CONTROL BOARD

UNDERGROUND STORAGE TANK PERMIT APPLICATION - FORM A

COMPLETE THIS FORM FOR EACH FACILITY/SITE

#4106

MARK ONLY ONE ITEM: ☐ 1 NEW PERMIT ☒ 3 RENEWAL PERMIT ☐ 5 CHANGE OF INFORMATION ☐ 7 PERMANENTLY CLOSED SITE ☐ 2 INTERIM PERMIT ☐ 4 AMENDED PERMIT ☐ 6 TEMPORARY SITE CLOSURE

I. FACILITY/SITE INFORMATION & ADDRESS - (MUST BE COMPLETED)

DBA OR FACILITY NAME	NAME OF OPERATOR	
AMERICAN STORES CO. DATA CENTER		
ADDRESS	NEAREST CROSS STREET	PARCEL # (OPTIONAL)
6200 CLARK AVE.	DUBLIN BLVD	
CITY NAME	STATE / ZIP CODE	SITE PHONE # WITH AREA CODE
DUBLIN	CA 94568	(510) 833-6000

✔ BOX TO INDICATE: ☒ CORPORATION ☐ INDIVIDUAL ☐ PARTNERSHIP ☐ LOCAL-AGENCY DISTRICTS ☐ COUNTY-AGENCY* ☐ STATE-AGENCY* ☐ FEDERAL-AGENCY*

* If owner of UST is a public agency, complete the following: name of supervisor of division, section or office which operates the UST _____

TYPE OF BUSINESS	✔ IF INDIAN RESERVATION OR TRUST LANDS	# OF TANKS AT SITE	E. P. A. I. D. # (optional)
☐ 1 GAS STATION ☐ 2 DISTRIBUTOR ☐ 3 FARM ☐ 4 PROCESSOR ☒ 5 OTHER	☐	ONE	CAC000791080

EMERGENCY CONTACT PERSON (PRIMARY)		EMERGENCY CONTACT PERSON (SECONDARY) - optional	
DAYS: NAME (LAST, FIRST)	PHONE # WITH AREA CODE	DAYS: NAME (LAST, FIRST)	PHONE # WITH AREA CODE
McDONNELL, KENT	(510) 833-6290		
NIGHTS: NAME (LAST, FIRST)	PHONE # WITH AREA CODE	NIGHTS: NAME (LAST, FIRST)	PHONE # WITH AREA CODE
" "	(510) 833-8244		

II. PROPERTY OWNER INFORMATION - (MUST BE COMPLETED)

NAME	CARE OF ADDRESS INFORMATION		
AMERICAN STORES CO.			
MAILING OR STREET ADDRESS	✔ box to indicate: ☐ INDIVIDUAL ☐ LOCAL-AGENCY ☐ STATE-AGENCY		
709 EAST SOUTH TEMPLE	☒ CORPORATION ☐ PARTNERSHIP ☐ COUNTY-AGENCY ☐ FEDERAL-AGENCY		
CITY NAME	STATE	ZIP CODE	PHONE # WITH AREA CODE
SALT LAKE CITY	UT	34102	(801) 539-0112

III. TANK OWNER INFORMATION - (MUST BE COMPLETED)

NAME OF OWNER	CARE OF ADDRESS INFORMATION		
AMERICAN STORES CO.			
MAILING OR STREET ADDRESS	✔ box to indicate: ☐ INDIVIDUAL ☐ LOCAL-AGENCY ☐ STATE-AGENCY		
709 EAST SOUTH TEMPLE	☒ CORPORATION ☐ PARTNERSHIP ☐ COUNTY-AGENCY ☐ FEDERAL-AGENCY		
CITY NAME	STATE	ZIP CODE	PHONE # WITH AREA CODE
SALT LAKE CITY	UT	34102	(801) 539-0112

IV. BOARD OF EQUALIZATION UST STORAGE FEE ACCOUNT NUMBER - Call (916) 322-9669 if questions arise.

TY (TK) HQ 44- ☐☐☐☐☐☐

V. PETROLEUM UST FINANCIAL RESPONSIBILITY - (MUST BE COMPLETED) – IDENTIFY THE METHOD(S) USED

✔ box to indicate: ☒ 1 SELF-INSURED ☐ 2 GUARANTEE ☐ 3 INSURANCE ☐ 4 SURETY BOND ☐ 5 LETTER OF CREDIT ☐ 6 EXEMPTION ☐ 7 STATE FUND ☐ 8 STATE FUND & CHIEF FINANCIAL OFFICER LETTER ☐ 9 STATE FUND & CERTIFICATE OF DEPOSIT ☐ 10 LOCAL GOVT. MECHANISM ☐ 99 OTHER _____

VI. LEGAL NOTIFICATION AND BILLING ADDRESS

Legal notification and billing will be sent to the tank owner unless box I or II is checked.

CHECK ONE BOX INDICATING WHICH ABOVE ADDRESS SHOULD BE USED FOR LEGAL NOTIFICATIONS AND BILLING: I. ☒ II. ☐ III. ☐

THIS FORM HAS BEEN COMPLETED UNDER PENALTY OF PERJURY, AND TO THE BEST OF MY KNOWLEDGE, IS TRUE AND CORRECT

TANK OWNER'S NAME (PRINTED & SIGNATURE)	TANK OWNER'S TITLE	DATE MONTH/DAY/YEAR
ROBERT L LEWIS	DIR MAINFRAME SERV	11/24/97

LOCAL AGENCY USE ONLY

COUNTY #	JURISDICTION #	FACILITY #	
01	000	304106	12/1/97

LOCATION CODE - *OPTIONAL*	CENSUS TRACT # - *OPTIONAL*	SUPVISOR - DISTRICT CODE - *OPTIONAL*

THIS FORM MUST BE ACCOMPANIED BY AT LEAST (1) OR MORE PERMIT APPLICATION - FORM B, UNLESS THIS IS A CHANGE OF SITE INFORMATION ONLY.

OWNER MUST FILE THIS FORM WITH THE LOCAL AGENCY IMPLEMENTING THE UNDERGROUND STORAGE TANK REGULATIONS

FORM A (6-95)

STATE OF CALIFORNIA
STATE WATER RESOURCES CONTROL BOARD

UNDERGROUND STORAGE TANK PERMIT APPLICATION - FORM B

#4106

COMPLETE A SEPARATE FORM FOR EACH TANK SYSTEM.

MARK ONLY ONE ITEM: ☐ 1 NEW PERMIT ☒ 3 RENEWAL PERMIT ☐ 5 CHANGE OF INFORMATION ☐ 7 PERMANENTLY CLOSED ON SITE ☐ 2 INTERIM PERMIT ☐ 4 AMENDED PERMIT ☐ 6 TEMPORARY TANK CLOSURE ☐ 8 TANK REMOVED

DBA OR FACILITY NAME WHERE TANK IS INSTALLED:

I. TANK DESCRIPTION

COMPLETE ALL ITEMS – SPECIFY IF UNKNOWN

A. OWNER'S TANK I. D. # 2

B. MANUFACTURED BY: STOCKTON MFG. MODERN WELDING CO.

C. DATE INSTALLED (MO/DAY/YEAR) 1/27/88 +1-

D. TANK CAPACITY IN GALLONS: 2,000

II. TANK CONTENTS

IF A-1 IS MARKED, COMPLETE ITEM C.

A. ☐ 1 MOTOR VEHICLE FUEL ☐ 4 OIL ☒ 2 PETROLEUM ☐ 80 EMPTY ☐ 3 CHEMICAL PRODUCT ☐ 95 UNKNOWN

B. ☒ 1 PRODUCT ☐ 2 WASTE

C. ☐ 1a REGULAR UNLEADED ☒ 3 DIESEL ☐ 6 AVIATION GAS ☐ 1b PREMIUM UNLEADED ☐ 4 GASAHOL ☐ 7 METHANOL ☐ 1c MIDGRADE UNLEADED ☐ 5 JET FUEL ☐ 8 M85 ☐ 2 LEADED ☐ 99 OTHER (DESCRIBE IN ITEM D. BELOW)

D. IF (A.1) IS NOT MARKED, ENTER NAME OF SUBSTANCE STORED DIESEL C. A. S. #:

III. TANK CONSTRUCTION

MARK ONE ITEM ONLY IN BOXES A, B, AND C, AND ALL THAT APPLIES IN BOX D AND E

A. TYPE OF SYSTEM: ☒ 1 DOUBLE WALL ☐ 2 SINGLE WALL ☐ 3 SINGLE WALL WITH EXTERIOR LINER ☐ 4 SINGLE WALL IN A VAULT ☐ 5 INTERNAL BLADDER SYSTEM ☐ 95 UNKNOWN ☐ 99 OTHER

B. TANK MATERIAL (Primary Tank): ☐ 1 BARE STEEL ☐ 2 STAINLESS STEEL ☒ 3 FIBERGLASS ☐ 4 STEEL CLAD W/ FIBERGLASS REINFORCED PLASTIC ☐ 5 CONCRETE ☐ 6 POLYVINYL CHLORIDE ☐ 7 ALUMINUM ☐ 8 100% METHANOL COMPATIBLE W/FRP ☐ 9 BRONZE ☐ 10 GALVANIZED STEEL ☐ 95 UNKNOWN ☐ 99 OTHER

C. INTERIOR LINING OR COATING: ☐ 1 RUBBER LINED ☐ 2 ALKYD LINING ☐ 3 EPOXY LINING ☐ 4 PHENOLIC LINING ☐ 5 GLASS LINING ☐ 6 UNLINED ☒ 95 UNKNOWN ☐ 99 OTHER

IS LINING MATERIAL COMPATIBLE WITH 100% METHANOL? YES ___ NO ___

D. EXTERIOR CORROSION PROTECTION: ☐ 1 POLYETHYLENE WRAP ☐ 2 COATING ☐ 3 VINYL WRAP ☐ 4 FIBERGLASS REINFORCED PLASTIC ☐ 5 CATHODIC PROTECTION ☐ 91 NONE ☒ 95 UNKNOWN ☐ 99 OTHER

E. SPILL AND OVERFILL, etc. SPILL CONTAINMENT INSTALLED (YEAR) 1988 DROP TUBE YES X NO ___ STRIKER PLATE YES X NO ___ OVERFILL PREVENTION EQUIPMENT INSTALLED (YEAR) 1990 DISPENSER CONTAINMENT YES ___ NO X

IV. PIPING INFORMATION

CIRCLE A IF ABOVE GROUND OR U IF UNDERGROUND, BOTH IF APPLICABLE

A. SYSTEM TYPE: A (U) 1 SUCTION A U 2 PRESSURE A U 3 GRAVITY A U 4 FLEXIBLE PIPING A U 99 OTHER

B. CONSTRUCTION: A U 1 SINGLE WALL A (U) 2 DOUBLE WALL A U 3 LINED TRENCH A U 95 UNKNOWN A U 99 OTHER

C. MATERIAL AND CORROSION PROTECTION: A U 1 BARE STEEL A U 2 STAINLESS STEEL A U 3 POLYVINYL CHLORIDE (PVC) A (U) 4 FIBERGLASS PIPE A U 5 ALUMINUM A U 6 CONCRETE A U 7 STEEL W/ COATING A U 8 100% METHANOL COMPATIBLE W/FRP A U 9 GALVANIZED STEEL A U 10 CATHODIC PROTECTION A U 95 UNKNOWN A U 99 OTHER

D. LEAK DETECTION: ☐ 1 MECHANICAL LINE LEAK DETECTOR ☐ 2 LINE TIGHTNESS TESTING ☒ 3 CONTINUOUS INTERSTITIAL MONITORING ☐ 4 ELECTRONIC LINE LEAK DETECTOR ☐ 5 AUTOMATIC PUMP SHUTDOWN ☐ 99 OTHER

V. TANK LEAK DETECTION

☐ 1 VISUAL CHECK ☒ 2 MANUAL INVENTORY RECONCILIATION ☐ 3 VADOZE MONITORING ☒ 4 AUTOMATIC TANK GAUGING ☐ 5 GROUND WATER MONITORING ☐ 6 ANNUAL TANK TESTING ☐ 7 CONTINUOUS INTERSTITIAL MONITORING ☐ 8 SIR ☐ 9 WEEKLY MANUAL TANK GAUGING ☐ 10 MONTHLY TANK TESTING ☐ 95 UNKNOWN ☐ 99 OTHER

VI. TANK CLOSURE INFORMATION (PERMANENT CLOSURE IN-PLACE)

1. ESTIMATED DATE LAST USED (MO/DAY/YR)

2. ESTIMATED QUANTITY OF SUBSTANCE REMAINING _____ GALLONS

3. WAS TANK FILLED WITH INERT MATERIAL? ☐ YES ☐ NO

THIS FORM HAS BEEN COMPLETED UNDER PENALTY OF PERJURY, AND TO THE BEST OF MY KNOWLEDGE, IS TRUE AND CORRECT

TANK OWNER'S NAME (PRINTED & SIGNATURE) ROBERT L LEWIS Robert L Lewis DATE 11/24/97

LOCAL AGENCY USE ONLY

THE STATE I.D. NUMBER IS COMPOSED OF THE FOUR NUMBERS BELOW

STATE I.D.#	COUNTY #	JURISDICTION #	FACILITY #	TANK #	
	01	000	304106	000002	12/1/97

PERMIT NUMBER	PERMIT APPROVED BY/DATE	PERMIT EXPIRATION DATE

THIS FORM MUST BE ACCOMPANIED BY A PERMIT APPLICATION - FORM A, UNLESS A CURRENT FORM A HAS BEEN FILED. FORM C MUST BE COMPLETED FOR INSTALLATIONS. THIS FORM SHOULD BE ACCOMPANIED BY A PLOT PLAN. FILE THIS FORM WITH THE LOCAL AGENCY IMPLEMENTING THE UNDERGROUND STORAGE TANK REGULATIONS

FORM B (6-95)

white -env.health
yellow -facility
pink -files

ALAMEDA COUNTY, DEPARTMENT OF ENVIRONMENTAL HEALTH

Hazardous Materials Inspection Form

80 Swan Way, #200
Oakland, CA 94621
(415) 271-4320

II,III

Site ID # 406 Site Name Lucky Store Today's Date 12/28/93

Site Address 6300 Clark Ave

City Dublin Zip 94568 Phone _____

____ MAX AMT stored > 500 lbs, 55 gal., 200 cft.?

Inspection Categories:
____ I. Haz. Mat/Waste GENERATOR/TRANSPORTER
____ II. Business Plans, Acute Hazardous Materials
__X__ III. Underground Tanks Removal

* Calif. Administration Code (CAC) or the Health & Safety Code (HS&C)

II.A BUSINESS PLANS (Title 19)

__	1. Immediate Reporting	2703
__	2. Bus. Plan Stds.	25503(b)
__	3. RR Cars > 30 days	25503.7
__	4. Inventory Information	25504(a)
__	5. Inventory Complete	2730
__	6. Emergency Response	25504(b)
__	7. Training	25504(c)
__	8. Deficiency	25505(a)
__	9. Modification	25505(b)

II.B ACUTELY HAZ. MAT'LS

__	10. Registration Form Filed	25533(a)
__	11. Form Complete	25533(b)
__	12. RMPP Contents	25534(c)
__	13. Implement Sch. Req'd? (Y/N)	____
__	14. OffSite Conseq. Assess.	25524(c)
__	15. Probable Risk Assessment	25534(d)
__	16. Persons Responsible	25534(g)
__	17. Certification	25534(f)
__	18. Exemption Request? (Y/N)	25536(b)
__	19. Trade Secret Requested?	25538

III. UNDERGROUND TANKS (Title 23)

General

__	1. Permit Application	25284 (H&S)
__	2. Pipeline Leak Detection	25292 (H&S)
__	3. Records Maintenance	2712
__	4. Release Report	2651
__	5. Closure Plans	2670

Monitoring for Existing Tanks

__ 6. Method
1) Monthly Test
2) Daily Vadose
Semi-annual gndwater
One time soils
3) Daily Vadose
One time soils
Annual tank test
4) Monthly Gndwater
One time soils
5) Daily Inventory
Annual tank testing
Cont pipe leak det
Vadose/gndwater mon.
6) Daily Inventory
Annual tank testing
Cont pipe leak det
7) Weekly Tank Gauge
Annual tank tstng
8) Annual Tank Testing
Daily Inventory
9) Other _____

__	7. Precis Tank Test Date: _____	2643
__	8. Inventory Rec.	2644
__	9. Soil Testing.	2646
__	10. Ground Water.	2647

New Tanks

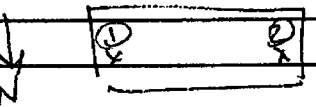
__	11. Monitor Plan	2632
__	12. Access. Secure	2634
__	13. Plans Submit Date: _____	2711
__	14. As Built Date: _____	2635

Rev 6/88

Comments:

LEL - 3%, O_2 19%
1K Fiberglass UST for diesel
Hauler: Trident.

Tanks in good condition. No holes or cracks.



① Brown sandy clay - No petroleum odor
Sample from 9' depth

② Sandy soil, no obvious odor
Sample from ~ 9' depth

II, III

Contact: _____

Title: _____ Inspector: Elva CHW

Signature: _____ Signature: _____

Ac-19

RAMCON

Engineering & Environmental Contracting, Inc.

RAMCON FAX COVER SHEET

DATE: 12-29-93
TO: Eva Chu
COMPANY: ALameda County
FAX NUMBER: 510 - 569 - 4757
FROM: Frank Pile
RAMCON FAX NUMBER: (916) 372-4209

COMMENTS

Eva,

Here is the analytical data on the soil samples taken from the tank we removed at lucky's Head Quarters there in Dublin At 6300 Clark Ave. ALL data is NON Detect. It is our intention to backfill that excavation on Monday the 3RD, if you need ANY further information please call me at numbers shown below

Thanks
Frank Pile

TOTAL NO. OF PAGES 9 (INCLUDING COVER SHEET)
DOCS WILL NOT BE FOLLOWED UP BY MAIL/FED X/COURIER.

IF YOU DO NOT RECEIVE IN LEGIBLE FORM: CALL (916) 372-7535

3751 COMMERCE DRIVE, WEST SACRAMENTO, CA 95691

December 29, 1993
Sample Log 8242

Frank Pile
Ramcon Engineering & Environmental Contracting, Inc.
P.O. BOX 1026
West Sacramento, CA 95691

Subject: Analytical Results for 2 Soil Samples
Identified as: Project # 586001 (Lucky's)
Received: 12/28/93
Purchase Order: 9338

Dear Mr. Pile:

Analysis of the sample(s) referenced above has been completed. This report is written to confirm results communicated on December 29, 1993 and describes procedures used to analyze the samples.

Sample(s) were received in brass sleeves that were sealed with PTFE sheets and plastic endcaps. Each sample was transported and received under documented chain of custody and stored at 4 degrees C until analysis was performed.

Sample(s) were analyzed using the following method(s):

"BTEX" (EPA Method 8020/Purge-and-Trap)
"TPH as Diesel,Motor Oil,Jet/Kerosene" (Mod. 8015/Extraction)

Please refer to the following table(s) for summarized analytical results and contact us at 916-757-4650 if you have questions regarding procedures or results. The chain-of-custody document is enclosed.

Approved by:

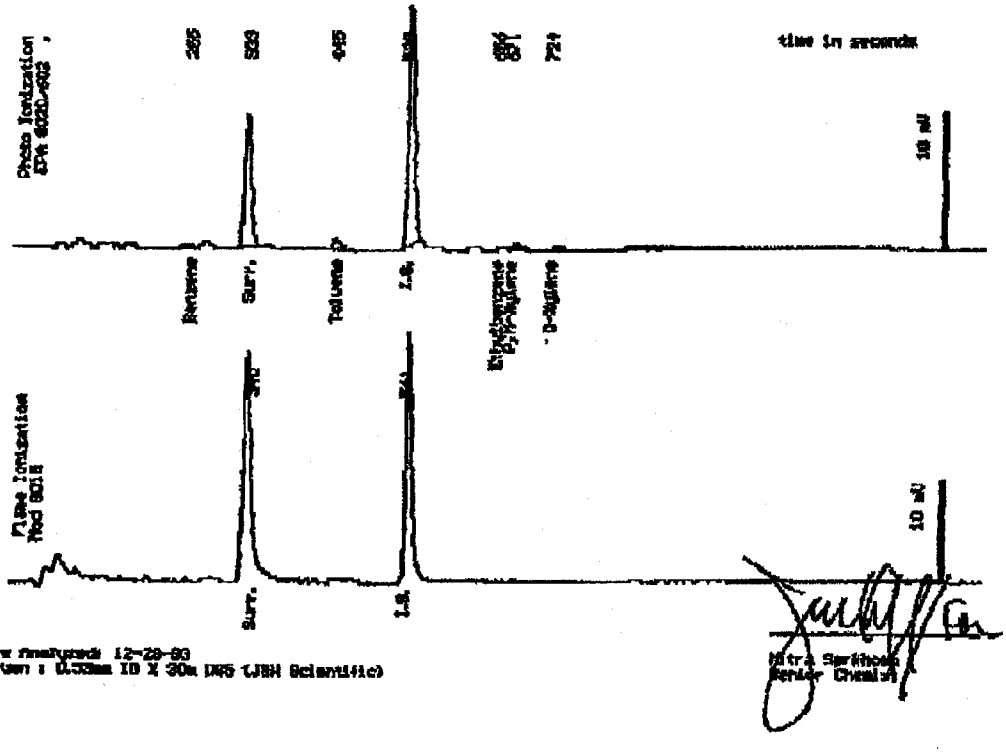
Stewart Podolsky
Senior Chemist

Sample Log 8242

Sample: PF-1

From : Project # 588001 (Lucky's)
Sampled : 12/28/93
Dilution : 1:1 QC Batch : 6076a
Matrix : Soil

Parameter	(MRL) mg/kg	Measured Value mg/kg
Benzene	(.0050)	<.0050
Toluene	(.0050)	<.0050
Ethylbenzene	(.0050)	<.0050
Total Xylenes	(.0050)	<.0050
Surrogate Recovery		97 %



Date Analyzed: 12-29-93
Column : 0.53mm ID X 30m DB5 (J&W Scientific)

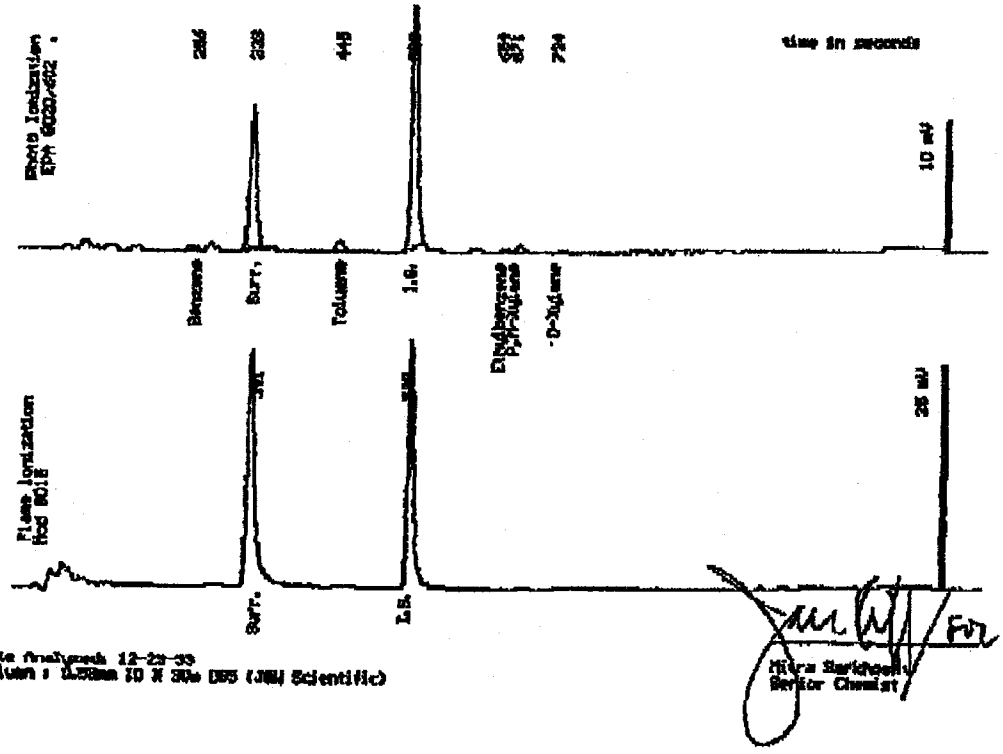
Mitra Kariotis
Senior Chemist

Sample Log 8242

Sample: PF-2

From : Project # 588001 (Lucky's)
Sampled : 12/28/93
Dilution : 1:1 QC Batch : 6076a
Matrix : Soil

Parameter	(MRL) ug/kg	Measured Value ug/kg
Benzene	(.0050)	<.0050
Toluene	(.0050)	<.0050
Ethylbenzene	(.0050)	<.0050
Total Xylenes	(.0050)	<.0050
Surrogate Recovery		101 %



Date Analyzed: 12-29-93
Column : 0.53mm ID X 30m DB5 (J&W Scientific)

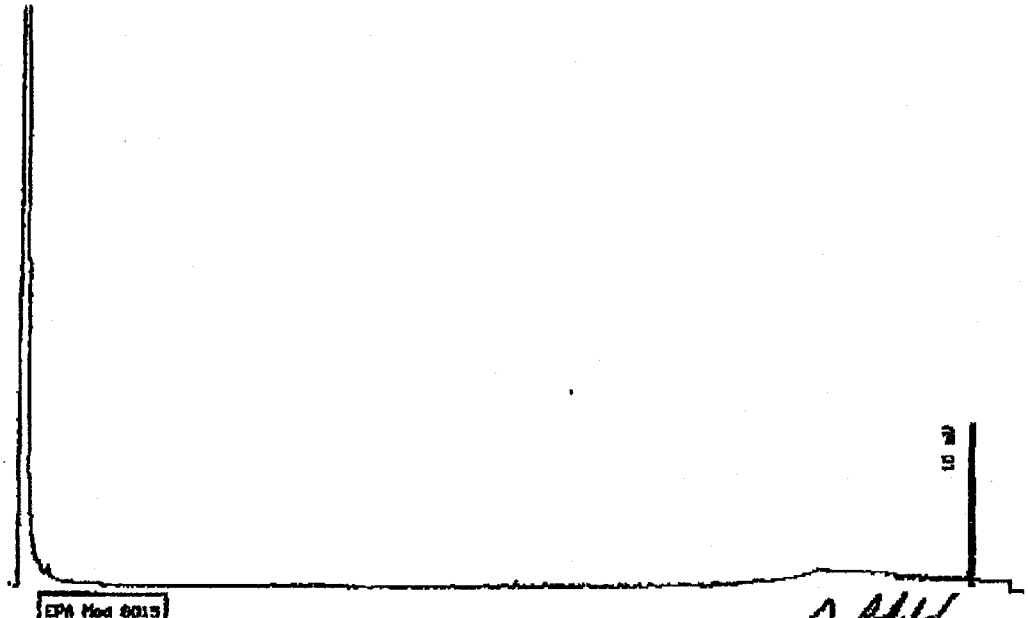
Mitra Barkhordar
Senior Chemist

Sample Log 8242

Sample: PF-1

From : Project # 588001 (Lucky's)
Sampled : 12/28/93
Extracted: 12/28/93 QC Batch : DS931211
Dilution : 1:1 Run Log : 8142B
Matrix : Soil

Parameter	(MDL) mg/kg	Measured Value mg/kg
TPH as Diesel	(10)	<10
TPH as Motor Oil	(10)	<10



Date: 12-28-93 Time: 23:59:31
Column : 0.53mm ID X 15m DB1 (J&W Scientific)

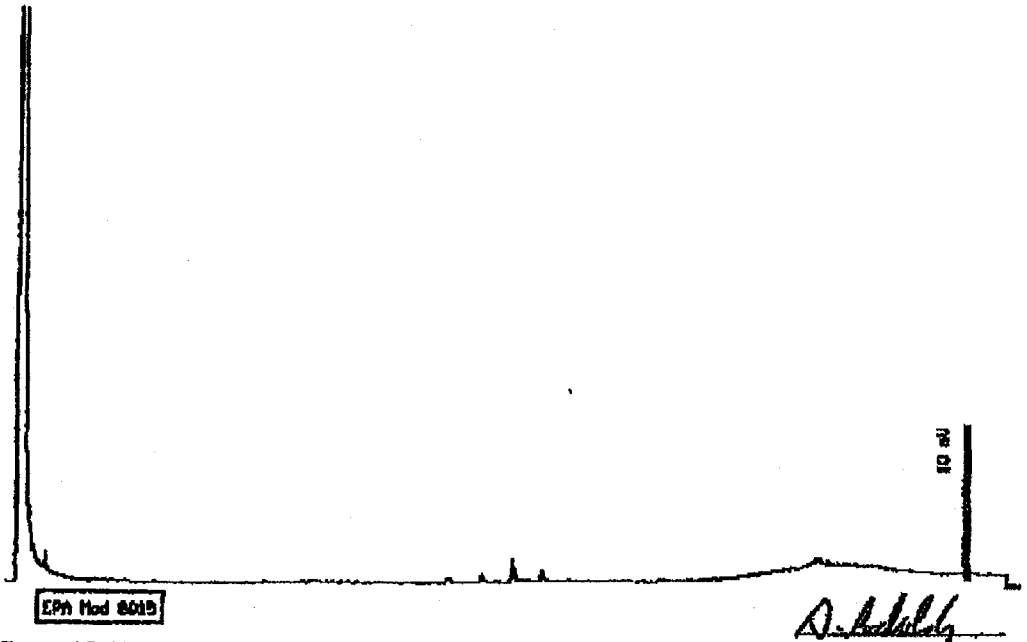
Stewart Podolsky
Senior Chemist

Sample Log 8242
8242-2

Sample: PF-2

From : Project # 588001 (Lucky's)
Sampled : 12/28/93
Extracted: 12/28/93 QC Batch : DS931211
Dilution : 1:1 Run Log : 8142B
Matrix : Soil

Parameter	(MDL) mg/kg	Measured Value mg/kg
TPH as Diesel	(10)	<10
TPH as Motor Oil	(10)	<10



Date: 12-28-93 Time: 22:10:55
Column : 0.53mm ID X 15m DB1 (J&W Scientific)

Eleanore Podolsky
Senior Chemist

Western Environmental Science & Technology
1046 Olive Drive, Suite 3
Davis, CA 95616
916-753-9500
FAX #: 916-753-9091
LAB #: 916-757-4859

CHAIN-OF-CUSTODY RECORD AND ANALYSIS REQUEST

Project Manager: FRANK PILE
Phone #: 372-7535
Company/Address:
FAX #:
Project Number: 508001
P.O.#: 7338
Project Name: LUCKY'S
Project Location: 6300 CLARK AVE DUBLIN CA.
Sampler Signature:

Sample ID	Sampling DATE	Sampling TIME	Container VOA	Container SLEEVE	Container 1L GLASS	Container 1L PLASTIC	Method Preserved HCl	Method Preserved HNO3	Method Preserved ICE	Method Preserved NONE	Matrix WATER	Matrix SOIL	ANALYSIS REQUEST: BTEX (8020/8021)	BTEX/TPH as Gasoline (8020/8015)	TPH as Diesel/Kerosene (8015)	Total Oil & Grease (5520 B&F)	Total Oil & Grease (5520 BVE,F,C)	96-Hour Fish Bioassay	EPA 601/8010	EPA 602/8020	EPA 8150/8150	EPA 8080/8080 - Pesticides	EPA 608/8080 - PCBs	EPA 624/8240	EPA 625/8270	ORGANIC LEAD	Reactivity, Corrosivity, Ignitability	CAM - 17 Metals	EPA - Priority Pollutant Metals	LEAD (7420/7421/239.2)	Cd, Cr, Pb, Zn, Ni	W.E.T. (-)	TOTAL (-)	TAT: RUSH SERVICE (24 hr) or (48 hr)	EXPEDITED SERVICE (48 hr) or (1 wk)	STANDARD SERVICE (2wk)
PC-1	12/28/93	11:45		1					X			X	X	X	X																				X		
PC-2	12/28/93	11:45		1					X			X	X	X	X																				X		

Relinquished by:	Date	Time	Received by:
Relinquished by	Date	Time	Received by:
Relinquished by	Date	Time	Received by Laboratory:
	12/28/93		

Remarks:
LAT 3:20

RECEIVED by W.E.S.T.

Bill To:

RAMCON / FRANK PILE 12-28-93

6300 CLARK AVE DUBLIN CA. (LUCKY'S) 025

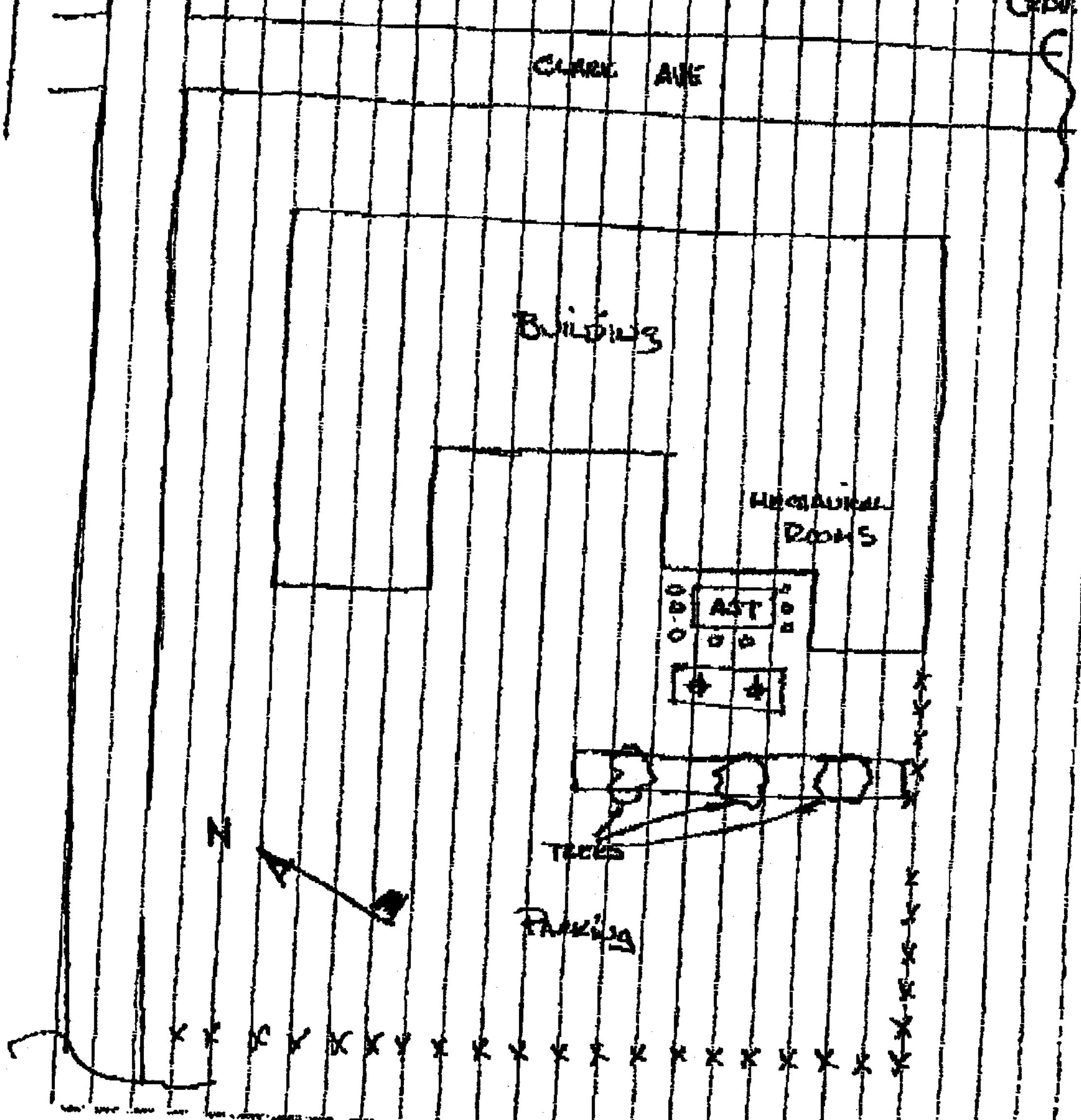
Pg 1 of 2

Project # 588001
PO # 9338

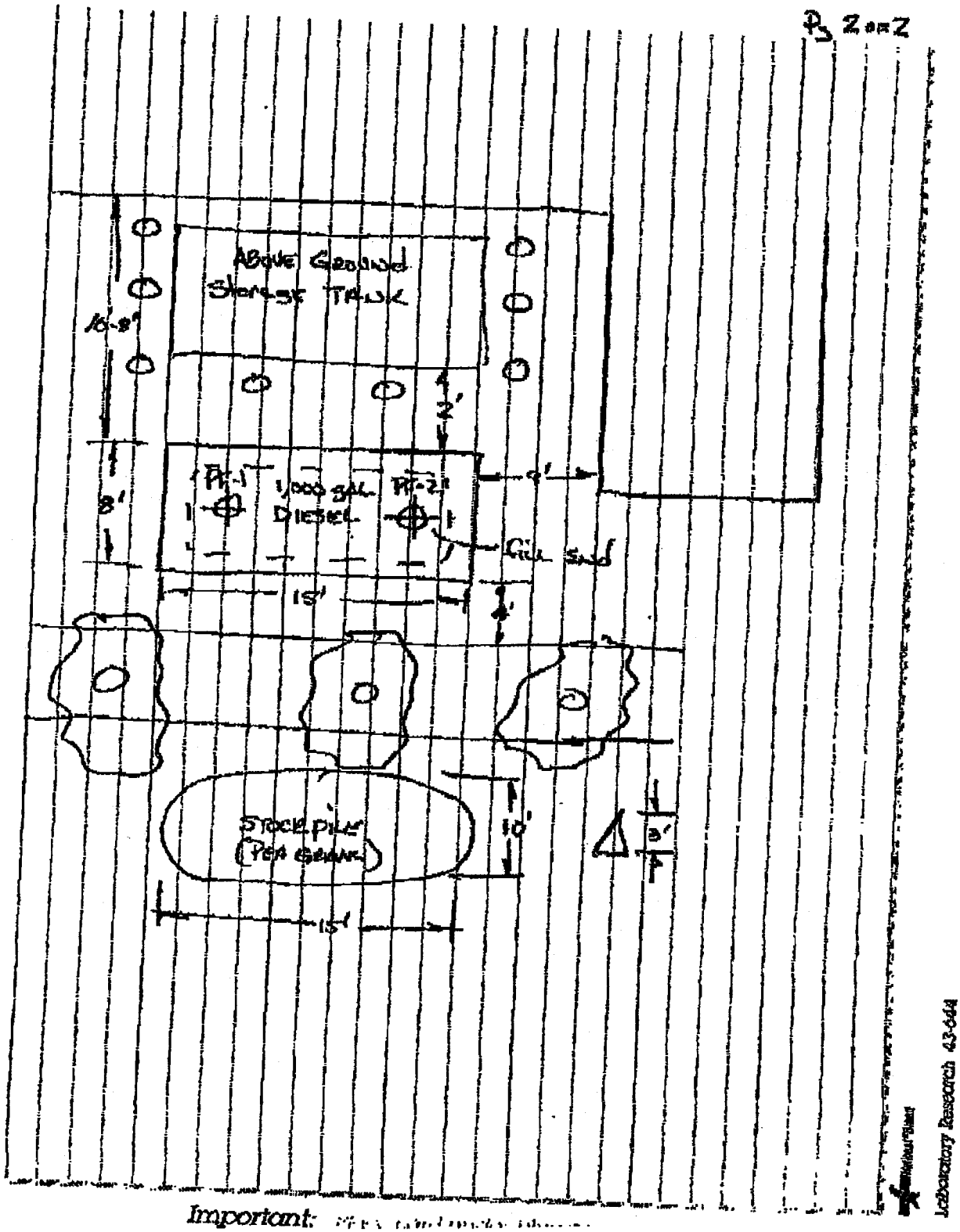
ARRIVAL : 13:45
Depart : 14:55
LFT : 3:20

PF-1, PF-2 were taken 9' Below grade
Packed Ø head space covered w/ Teflon
Capped and put on Ice for transport
2x6" Brass sleeves.

Present: C. Goodrich (Lucky)
B. Edmiston (Ramcon)
E. Chu (Alameda Co. Env. Health)



026



Important:

Analysis of MTBE and BTEX by Direct Aqueous Injection GC/MS

Steven M. Pyle

United States Environmental Protection Agency, National Exposure Research Laboratory
Environmental Sciences Division, P.O. Box 93478, Las Vegas, NV 89193-3478, USA

EPA

1 ABSTRACT

A direct aqueous injection (DAI) method was developed for the determination of the fuel oxygenate, methyl tert-butyl ether (MTBE), along with benzene, toluene, ethyl benzene, and xylene (BTEX) and several other alkylated benzenes. These compounds are commonly found in contaminated groundwater due to leaking underground gasoline storage tanks. Methanolic stock solutions of these compounds (plus nine other volatile components) were spiked into distilled water at the 20- to 20,000-ppb levels and analyzed by direct aqueous sample introduction into a fused-silica capillary column interfaced to a benchtop ion trap mass spectrometer (GC/MS). Using the method of external standardization, the response factors, retention times, concentration range, and method detection limits for the 14 compounds were determined. Replicate data (n=7) was collected at each concentration and precision data (%RSD) generated. For comparison, matched detection limits (MDLs) were determined from the data by three commonly used methods.

Replicate injections by DAI of a 10-ppm solution over a 16 hour time period (n=24) were used to determine the concentration decay in water of the 14 constituents in an open container at room temperature.

2 INTRODUCTION

Methyl tert-butyl ether (MTBE) is a "fuel oxygenate", a chemical added to motor fuels primarily for the purpose of improving fuel combustion and reducing emissions such as carbon monoxide and other pollutants. MTBE can find its way into groundwater by a variety of means, often from leaking underground fuel tanks. Its toxicology is poorly understood. While volatile, MTBE is also miscible with water and therefore poorly purgeable. It is therefore not amenable to the Agency's normal chemical analysis methods. We have developed a method for the direct aqueous injection (DAI) analysis of poorly purgeable pollutants, as well as other volatile and semivolatile components present in gasoline-contaminated groundwater. This technique uses direct aqueous sample injection into a gas chromatograph/mass spectrometer for qualitative and quantitative analysis. DAI is rapid, sensitive, easily applied, and generates no waste solvent.

3 EXPERIMENTAL

Standard Solutions

Standard and stock solutions were prepared from 2 commercially available methanolic Supelco Standards, Methyl tert-Butyl Ether (catalog # 4-8483) and Volatile Organic Compounds Mix 2 (catalog # 4-8777). A 10-µL syringe was used to add the appropriate volume directly through the septum into an inverted 1.5-mL autosampler vial containing 1.0 mL of distilled water (see Table 1). The lower concentrations were prepared from a 1 to 100 dilution of the standards. Aqueous standards were run within 10 hours of preparation.

Table 1. Preparation of aqueous standards.

final concentration (ppb)	initial concentration (µg/mL)	volume added (µL)
20	20*	1
200	20*	10
2 000	2 000	1
10 000	2 000	5
20 000	2 000	10

* 1/100 dilution of initial standard solution

Conditions

After some initial experimentation, the following conditions were used to collect the data for method development.

GC Conditions

initial temperature	40 °C
initial time	1 min
temperature rate	10 °C/min
final temperature	240 °C
final hold time	6 min
total run time	21 min
transfer line	240 °C

SPI Injection

initial temperature	60 °C
initial time	0.1 min
temperature rate	150 °C/min
final temperature	280 °C
final hold time	18 min
total run time	19.56 min
injection volume	0.5 µL (delivered by autosampler)

Mass Spectrometer

scan range	40 to 300 amu
scan time	0.6 sec/scan
mass defect	0 mmu/100 amu
acquire time	20 min
solvent delay	2 min
background mass	45 m/z

Column

dimensions	25 m × 0.20 mm × 0.5 µm film
liquid phase	5% diphenyl-95% dimethyl polysiloxane
head pressure	10 psig helium
linear velocity	35 cm/sec at 40 °C

4 CALCULATIONS

Method Detection Limit Calculation #1 (MDL1)

This EPA formulated MDL calculation is based on a statistical argument (1,2) and is defined as the minimum concentration of a substance greater than zero that can be measured with 99% confidence. It is calculated from the formula:

MDL = (% rsd × 3.143 × concentration)

where % rsd is the relative standard deviation in per cent, and 3.143 Student's *t* value which, in this case, is for 7 replicate injections. The method stipulates that the concentration of the replicates must not be greater than 5 times the resulting calculated MDL.

Method Detection Limit Calculation #2 (MDL2)

This calculation (3) is a quick and simple estimate of the MDL based on the assumption that the minimum area count that is discernible from background is the same for any analyte. It uses the formula:

MDL = (area discernible from background ÷ response factor)

where the area discernible from background is defined as the area of a peak that is three times the noise level. Response factor is the average peak area over the linear range per amount (in this case in picograms injected on-column).

Method Detection Limit Calculation #3 (MDL3)

This method detection limit calculation (4) is determined from standard injections. In this case a 10-ppm standard. The S/N was determined for each analyte's quantitation ion using the GC/MS software and extrapolated down to a S/N of 10. This approach purportedly gives more realistic MDLs for the DAI-GC/MS than the MDL1 method above.

Concentration Decay in Water Calculation

A logarithmic half-life decay model did not fit the data sufficiently to determine a half-life so a linear model was used to calculate the rate of concentration decay with time. A least squares regression analysis was applied to the time (independent variable) and concentration (dependent variable) data and the slope used as a measure of decay reported in Table 2 as units of ppb/min.

Table 2. Compound number, quantitation ion, retention time, response factor, %RSD, MDLs, decay rate, and water solubility (5).

no.	compound	quan ion (m/z)	retention time (min)	resp factor (area/pg)	RSD (%)	MDL1 (ppb)	MDL2 (ppb)	MDL3 (ppb)	decay rate (ppb/min)	solubility (ppm)
1	Methyl-t-butyl ether	73	3.09	10.2	14	12	26	6	-11.46	51000
2	Benzene	78	3.99	15.2	10	21	11	11	-11.09	1800
3	Methylbenzene	91	4.46	22.7	11	6	8	20	-10.35	526
4	Ethylbenzene	91	6.29	21.0	13	7	10	20	-9.50	206
5	m-Xylene	91	6.37	20.8	13	9	8	76	-9.54	----
6	Styrene	104	7.02	10.9	12	18	17	40	-9.67	320
7	Bromobenzene	158	7.49	7.4	15	9	26	5	-9.19	----
8	1,3,5-Trimethylbenzene	105	8.28	19.3	11	6	9	42	-6.27	----
9	1,2,4-Trimethylbenzene	105	9.57	18.8	13	6	11	31	-6.40	----
10	p-Isopropyltoluene	119	9.20	15.8	11	6	10	20	-4.62	----
11	n-Butylbenzene	91	10:04	16.6	14	14	9	52	-6.02	----
12	1,2,4-Trichlorobenzene	180	12.14	8.4	13	11	18	127	-4.23	49
13	Naphthalene	128	12:24	33.7	9	8	5	64	-6.66	31
14	1,2,3-Trichlorobenzene	180	12:52	9.1	11	9	15	48	-4.46	----

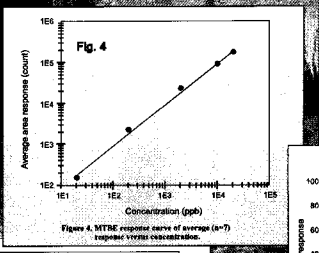


Figure 4. MTBE response curve of average (n=7) response versus concentration.

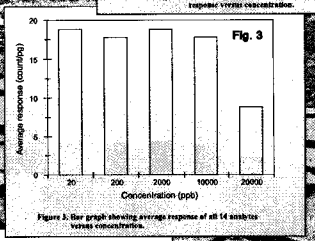


Figure 3. Bar graph showing average response of all 14 analytes versus concentration.

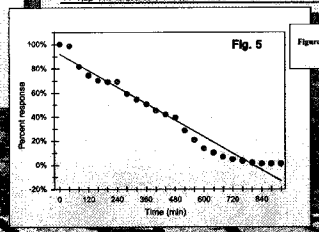


Figure 5. Concentration decay of MTBE with time.

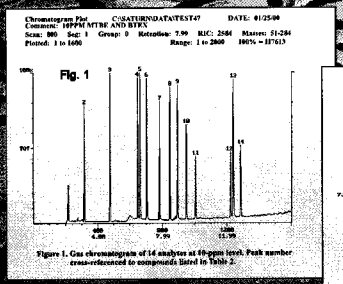


Figure 1. Gas chromatogram of 14 analytes at 10-ppm level. Peak number cross-referenced to compounds listed in Table 2.

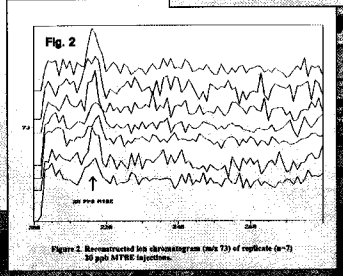


Figure 2. Reconstructed ion chromatogram (m/z 73) of replicate (n=7) 20 ppb MTBE injections.

5 CONCLUSIONS

1) DAI analysis showed good chromatographic separation and peak shape.
2) Adequate sensitivity (20 ppb) and precision (average of 12% RSD at 200 ppb level) was obtained for MTBE and 13 other components.
3) DAI was applicable over a 3-decade concentration range.
4) DAI is rapid, easily applied, and generates no waste solvent.
5) Application of DAI to concentration versus time showed 50% of volatiles were lost in average of approximately 500 minutes under quiescent conditions.

6 References

1) U.S. EPA Method, *Test Methods for Evaluating Solid Waste: Laboratory Manual. Physical/Chemical Methods. SW-846*. 3rd ed.; Washington, DC, 1986 U.S. EPA, November 1986.
2) Pyle, S. M. and A. B. Marcus. *International Journal of Environmental Analytical Chemistry*. 76(1). (2000).
3) Pyle, S. M. and D. F. Gurka. *Talanta*. 41. 1845-1852 (1994).
4) Church, C. D., L. M. Isabelle, J. F. Pankow, D. L. Rose, and P. G. Tratnyek. *Environ. Sci. Technol.* 31, 3723-3726 (1997). (http://cgr.ese.ogi.edu/mtbe/)
5) http://chemfinder.camsoft.com/

AC-19

tion, the following conditions were
hod development.

40 °C
1 min
10 °C/min
240 °C
0 min
21 min
240 °C

60 °C
0.1 min
150 °C/min
280 °C
18 min
19.56 min
0.5 µL (delivered by autosampler)

40 to 300 amu
0.6 sec/scan
0 mmu/100 amu
20 min
2 min
45 m/z

25 m × 0.20 mm × 0.5 µm film
5% diphenyl-
95% dimethyl polysiloxane
10 psig helium
35 cm/sec at 40 °C

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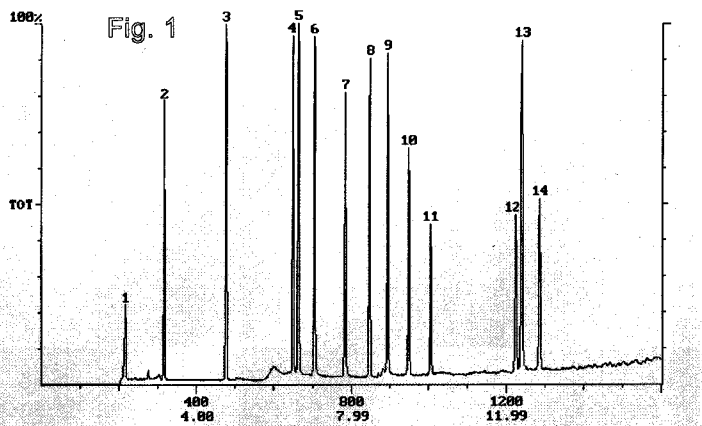
Chromatogram Plot C:\SATURN\DATA\TEST47 DATE: 01/25/00
Comment: 10PPM MTBE AND BTEX
Scan: 800 Seg: 1 Group: 0 Retention: 7.99 RIC: 2584 Masses: 51-284
Plotted: 1 to 1600 Range: 1 to 2000 100% = 117613



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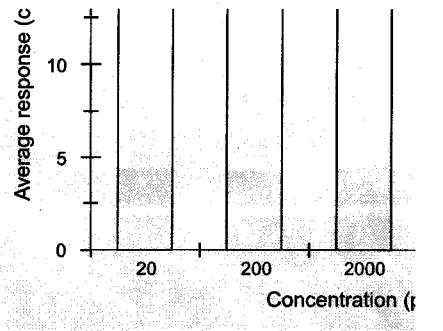

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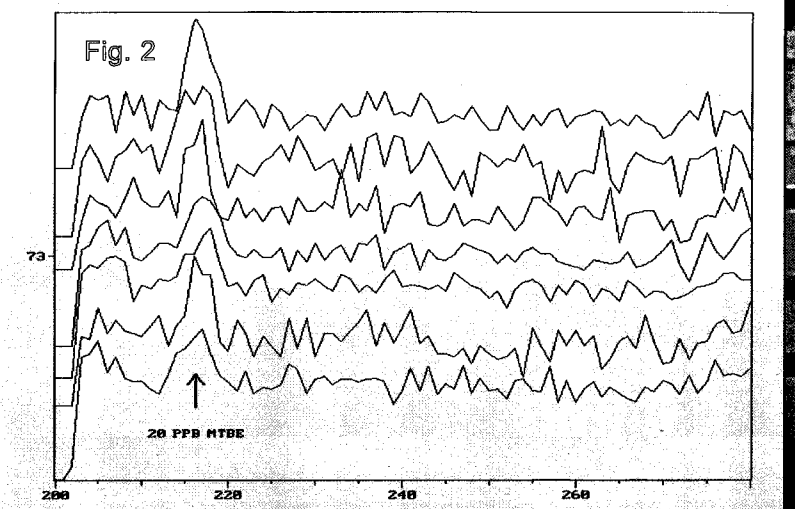

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5	m-Xylene	91	6:37	20.8	13	9	8	76	-9.54	----
6	Styrene	104	7:02	10.9	12	18	17	40	-9.67	320
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9	1,2,4-Trimethylbenzene	105	8:57	18.8	13	6	11	31	-6.40	----
10	p-Isopropyltoluene	119	9:29	15.8	11	6	10	20	-4.62	----
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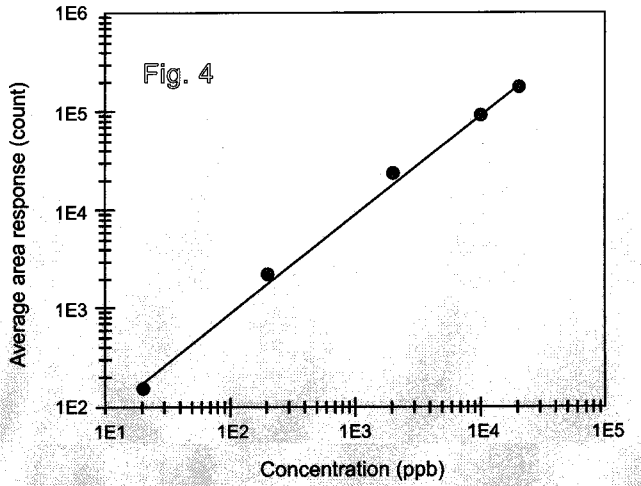

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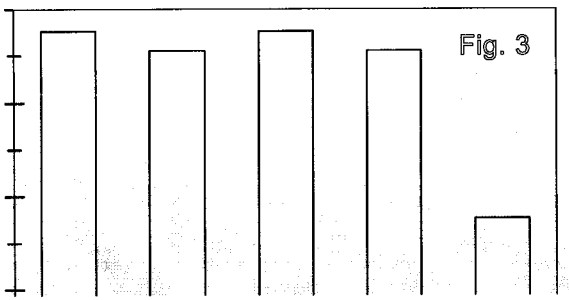

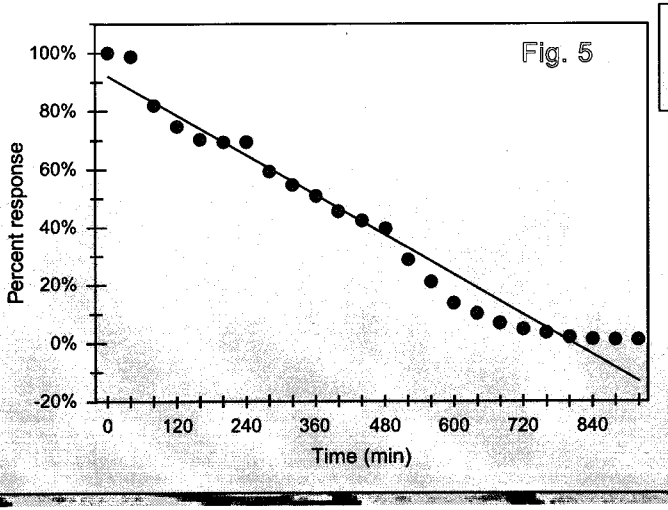

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