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**AQUEOUS PHASE LIQUID-LIQUID EXTRACTOR
(APLLE) 1983 PROJECT REPORT**

by

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Directorate**

**Direction Générale
des Eaux Intérieures**

Savile (9)

ES-553

Study 83 36-289(2216B)

**AQUEOUS PHASE LIQUID-LIQUID EXTRACTOR
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January 1984

ABSTRACT

This device is an upgraded version of two previous units and is designed to perform the first stage of extracting and concentrating, in the field, of organic compounds dissolved in natural waters for eventual analysis in the laboratory.

The unit mixes a large volume of water sample with a water insoluble organic fluid to remove the contaminants from the water and perform an initial concentration of about 200:1.

Further laboratory processing increases the overall concentration to about 200,000:1 and prepares the sample for analysis.

RÉSUMÉ

L'appareil décrit est une version améliorée des deux déjà existantes; il est conçu pour faire la première étape de l'extraction et de la concentration, sur le terrain, des composés organiques dissous dans les eaux naturelles afin d'en permettre l'analyse en laboratoire.

Cet appareil mélange un grand volume d'échantillons aqueux avec un solvant organique non miscible avec l'eau afin d'en extraire les contaminants et de faire une première concentration d'environ 200:1.

Ensuite, un traitement en laboratoire augmente la concentration globale à environ 200 000:1 et prépare l'échantillon pour l'analyse.

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1.0 INTRODUCTION

Organic materials in natural waters exist in extremely small quantities and in many forms. Both natural and man made organics are dispersed throughout almost all natural waters. Some of these materials are adsorbed strongly to clay size particles and may be removed and concentrated by centrifuging. The materials can then be stripped from the deposit by chemical means and analyzed. Some organics are not strongly adsorbed and remain in-solution in the water in very low concentration (parts per billion) yet some of these are very important to identify and quantify (dioxin, etc.).

The process of preferentially dissolving these materials into a very pure organic solvent having a low miscibility in water gives the possibility of a field usable technique to concentrate and extract the organics in a large volume of water into a small, easily handled, volume of solvent. Two previous units were built to explore the principle. One was of 200 litres capacity and the other of 20 litres. The first 200 l prototype has been extensively modified during its years of service. The second unit was specifically designed for use in areas where all equipment had to be hand carried for some distance and has been serving well with no changes. The MARK III unit has incorporated all the knowledge gained from the other two with some improvements.

2.0 DESIGN CRITERIA

Capacity of sample - 200 μ

Pump throughput - 45 L/min at 100 kPa

Solvent - methylene chloride (ultra high purity), 4.5 μ

Motor - Totally enclosed, fan cooled, continuous rated

Switch - Totally enclosed, motor starting

Construction materials - Brass, stainless steel, Teflon[®], glass, Kalrez[®], amorphous carbon, ceramic, aluminum alloy

Portability - Unit to be mounted on pneumatic casters, able to pass through standard doorways and to fit into domestic vans, lightweight, non-corrosive construction, minimal amounts of paint.

3.0 DISCUSSION

The MARK I APLE has been in use for some three years. It has some minor drawbacks, however, and some features found to be unnecessary. The volume of samples needing processing made another full size unit mandatory in 1983 and it was decided to make some design changes to freeze the design for future units.

In the MARK I unit an all "Teflon"® pump with variable speed drive was used, but since it was always operated at full speed to cut down processing time, a fixed speed pump was chosen. The teflon pump was deemed unnecessary, and a stainless steel pump with ceramic and carbon bearings, having a sealless magnetic drive was chosen. The only change required to the pump was the replacement of a "Viton"® O ring with one made of "Teflon"® as the methylene chloride causes most elastomers, including "Viton"®, to swell or degrade. This pump saved a premium of some 40% over the original.

A new, all aluminum frame was designed to be lower and wider than the original steel unit. This required no paint, which is advantageous, since methylene chloride is the prime ingredient in paint removers.

Large, pneumatic tired casters were fitted at the corners of the frame to increase stability and decrease rolling resistance and noise.

The centrifugal pump is located under the stainless steel drum, which is the main container of the system, and draws from the rear of the drum via a tee. The tee is also connected to a drain valve used to empty the methylene chloride and the processed water at the end of the cycle. The mixed fluids flow through the pump forwards and upwards to a spray bar located inside the top of the drum. The holes in the spray bar are deburred by glass bead blasting inside and out. On this bar (12.7 mm dia. tube) are located 4 rows of 1.6 mm diameter holes, spaced at 25 mm intervals, which when the fluid is

forced through them cause sufficient turbulence to intimately mix the water and methylene chloride in a very short time.

A glass tube sight gauge is mounted on the front cover behind a protective cage for judging the volume of the contents.

A 25.4 mm filler is located at top near the front and can be fitted with a stainless steel funnel (for filling) or a sealed cap (when in operation or in storage).

A drain line and valve are teed off at the exit of the pump and run back to the main drain. They are used to remove the last traces of solvent from inside the pump cavity and the spray bar supply line, after the main drain has been used.

The main drain valve is stainless steel and teflon construction internally with a brass body.

Atop the unit are a pair of relief valves set to about 30 kPa cracking pressure to vent any excessive internal pressure. These valves must be modified by replacing the "O" rings with ones of "Kalrez"® as no other elastomer will withstand the effects of methylene chloride.

The gaskets provided with the drum closures must also be replaced with units of "Teflon".

Wherever possible joints are made by welding or by "Swagelok"® tube fittings.

All portions of the unit must be cleaned extremely thoroughly before the unit is put into service. Initially all valves, fittings, the drum, and tubes are disassembled and solvent washed with ultra-high purity methylene chloride, followed by rinsing in similar purity isopropanol. All parts are then reassembled using graphite lubricant in isopropanol on external threads and "Teflon"® tape cleaned with UHP isopropanol on pipe threads. Aluminum foil protectors are fitted to all openings to keep the possibility of contamination to a minimum in the interval between cleaning and reassembly.

After assembly is complete 4.5 l of UHP methylene chloride are circulated through the system for 30 minutes and removed. This sample is then processed and checked in the normal way to assure that it is blank, that no contaminants remain in the system. The unit is then sealed and is ready for field use.

In the field the normal water sample is 200 l taken from the outlet of a centrifuge, particulates removed, to discriminate between adsorbed and dissolved contaminants. Once 200 l have been taken, 4.5 l of UHP methylene chloride are added, the unit sealed by removing the funnel and screwing down the cap, and the pump is started. The water and solvent mixture are circulated for about an hour and the pump is stopped. After at least 30 minutes undisturbed the solvent will normally have separated and sunk to the bottom of the system where it can be drained off into a sample bottle using the main and auxiliary drain valves. The water sample is then discarded either by draining through the main valve or by removing a cap on a tee at the front of the machine and pumping the system out. Post sample cleaning normally consists of pumping a few litres of solvent through the system and discarding it. In some natural waters with high organic contents, foaming, and the formation of a semi-stable emulsion may occur. Foaming can be minimized by filling the unit to above the spray bar and thereby minimizing the air available for entrainment. If a semi-stable emulsion occurs it will generally dissipate by leaving the unit overnight and draining it the following morning.

In the laboratory a second separation of water from solvent is done, the solvent is placed in a rotary vacuum evaporator and its volume is greatly reduced. The contaminants generally have a very much higher boiling point than the solvent and stay in the residue. Near the end of the evaporation a few mL of isopropanol are introduced. This has a slightly higher boiling point and allows the complete removal of the chlorinated solvent and the concentration of virtually all the contaminants into about 1 mL of carrier.

The overall concentration ratio thus becomes about 200,000:1, allowing a Gas Chromatograph/Mass Spectrometer to detect and analyze contaminants originally present in the parts per billion range.

Methylene chloride has a very low flamability but it should be handled carefully as it is very aggressive to paint, lubricants, rubber, and human skin.

In constructing this unit all welds were made by the tungsten, inert gas process, all tube fittings were pre-swaged, the drum was held to the frame by stainless steel bands and stainless steel fasteners were used throughout. Due to the aggressive nature of the solvent the thread lubricants applied to the tube fittings at the factory are removed by the cleaning process. These threads must be relubricated before assembly using molybdenum disulphide, graphite, or teflon powders, or metal to metal galling will occur, ruining the fitting and causing leaks.

A slightly modified version, MARK IIIB, has been designed to better suit the needs of some of the scientists and drawings of this version are included in the appendix.

4.0 CONCLUSIONS

This apparatus has proved its worth and is now in great demand. The design seems to have matured and should now be frozen in the main, though small modifications are of course possible at any time such as extra fill ports for continuous sampling over long time periods using a metering pump.



Fig.1. A.P.L.L.E. READY TO LOAD



Fig.2. A.P.L.L.E. READY TO RUN

APPENDIX I

DRAWING LIST

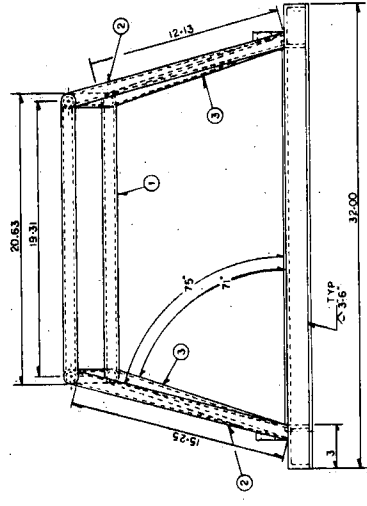
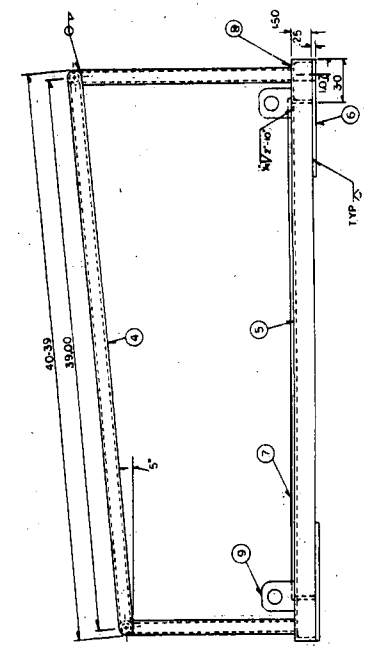
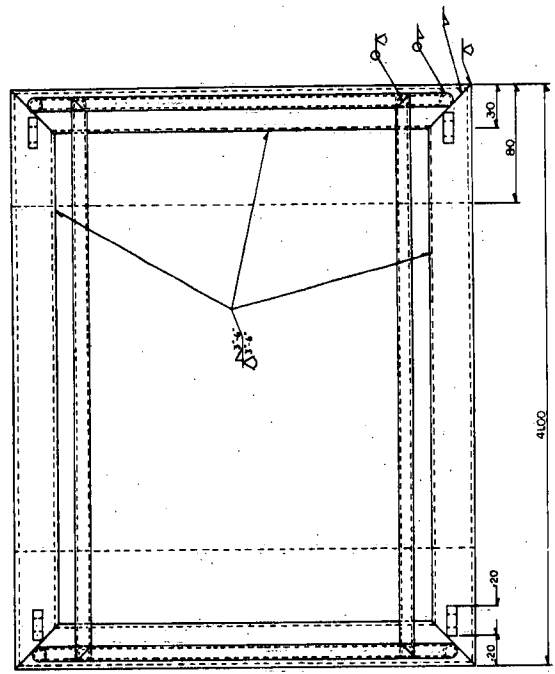
Drawing Number	Description
ME-5184-1	Frame Design
ME-5184-2	Filling Port
ME-5184-3	Plumbing Schematic
ME-5184-4	Sight Glass
ME-5184-5	Spray Bar
ME-5184-6	Cap and Washers
ME-5184-7	Funnel Extension
ME-5184-8	Plumbing Front View

PARTS LIST

PART NO.	DESCRIPTION	QTY
1	6061-T6 ALUM PIPE X 20 7/8 L	2
2	6061-T6 ALUM PIPE X 15 1/4 L	2
3	6061-T6 ALUM PIPE X 12 1/4 L	2
4	6061-T6 ALUM PIPE X 20 7/8 L	2
5	3017-T6 ALUM PIPE X 10 L	2
6	6061-T6 ALUM PLATE	1
7	3017-T6 ALUM PLATE	1
8	6061-T6 ALUM EX 32 L	2
9	LIFTING EYE 661-T6 ALUM	4

NOTE

1. DRILL VENT HOLES AT LOW POINTS OF ALL TUBES BEFORE WELDING.
2. DRILL CASTER AND PUMP MOUNTING HOLES TO SUIT ON ASSY.
3. SECURE DRUM WITH 3/8 SS. BANDIT IN TWO PLACES.
4. REMOVE ROLLING RINGS FROM DRUM IF FITTED.



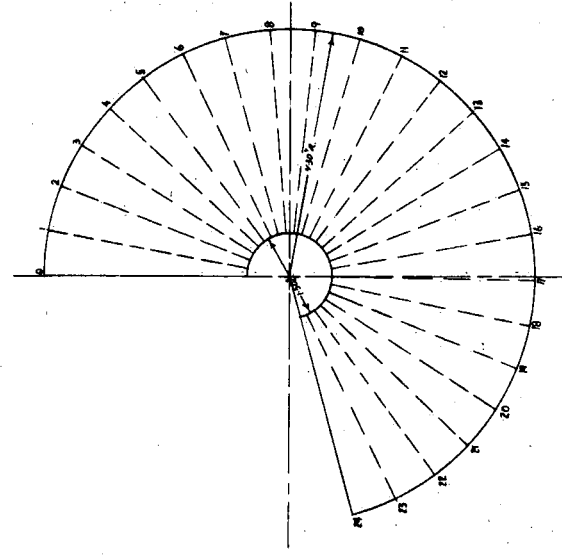
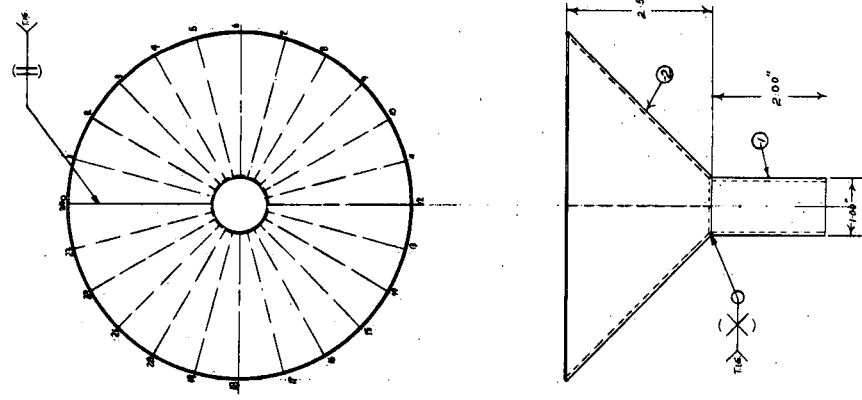
App. I - 1

	Development Design Check Control for Material for Material	Development Design Check Control for Material for Material
THE A-PILE MK III FRAME DESIGN		
CHECKED BY B. BAIN	APPROVED BY ME 5984-1	DRAWN BY B. BAIN
SHEET NO. 14	SHEET OF 26	PART OF

DATE	DESCRIPTION	BY	APP'D

5058 WELDING ROD 6061-T6
 5058 WELDING ROD 6061-T6

PARTS LIST	
ITEM NO.	DESCRIPTION
1	SPRING
2	1" x 3/16" W. 30-55 TUBES 1/2
3	20# 1/2" x 1/2" C.P.S.



App. I -2

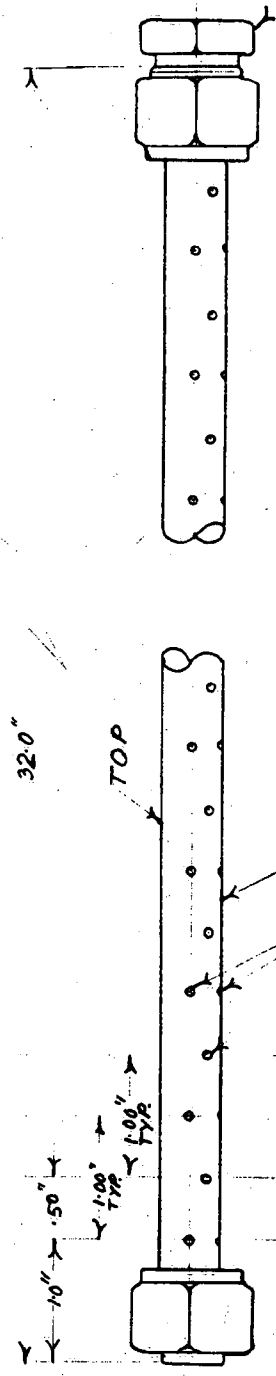
Development
 Conical
 Conical
 Conical
 for Metal Plates
 for Metal Plates

THE APP. SERIES, Pt. III
 FILING PART

CHECKED BY: [Signature]
 DATE: 11-15-52
 APPROVED BY: [Signature]
 DATE: 11-15-52

DATE	DESCRIPTION	BY

PARTS LIST	
PART NO.	DESCRIPTION
QTY.	



SS-810-C

3.00" O.D. X .035" W. TYPE 316 SS. TUBE

1/16" D. HOLES, 5 ROWS, 45° A PART, 10° CTRS., 5 STAGGER

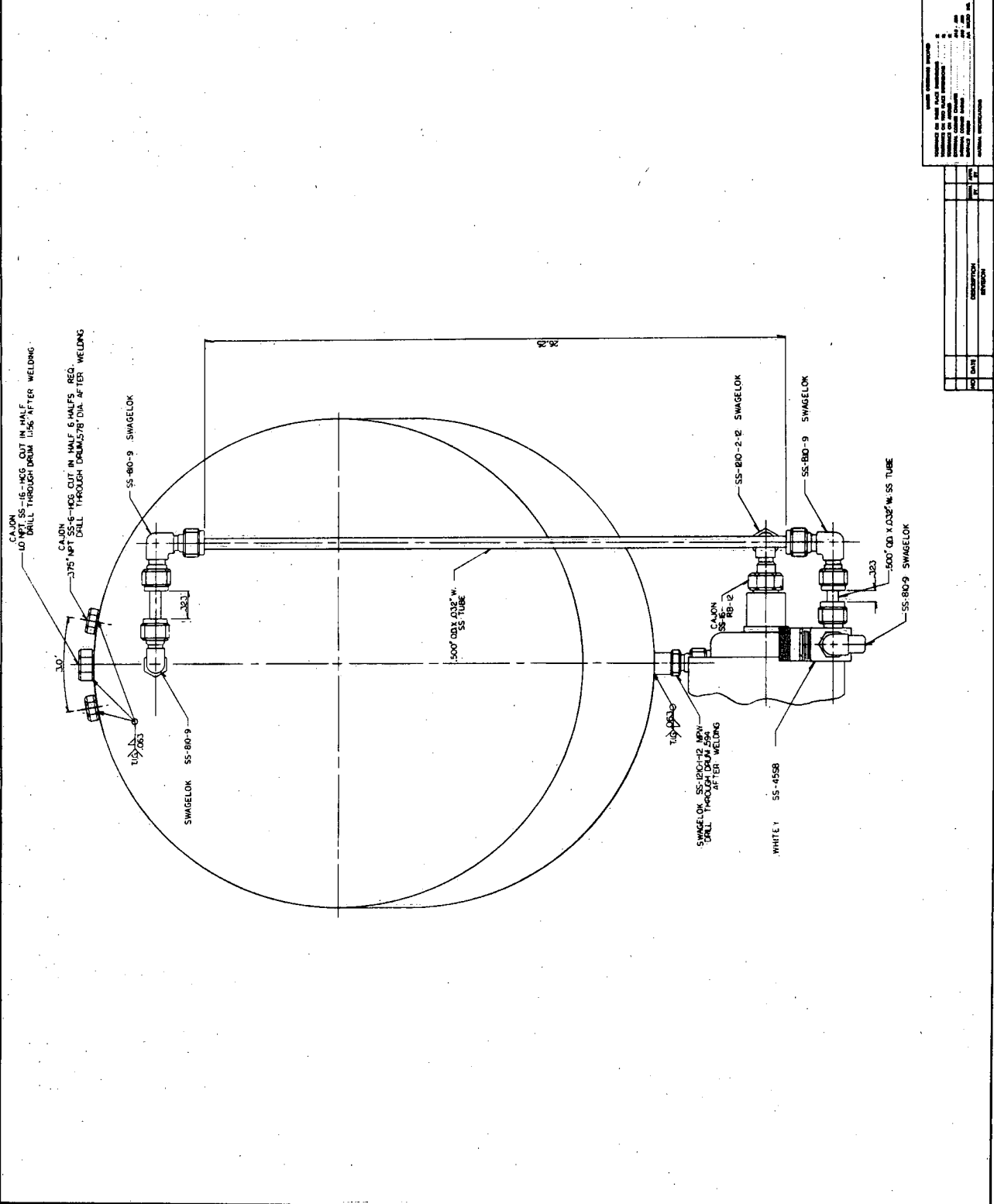
App. I -5

UNLESS OTHERWISE SPECIFIED	
TOLERANCE ON THREE PLACE DIMENSIONS	±
TOLERANCE ON TWO PLACE DIMENSIONS	±
TOLERANCE ON ANGLES	±
INTERNAL CORNER CHAMFER	.015 .025
INTERNAL CORNER RADIUS	.010 .020
SURFACE FINISH	AA MICRO INS.
MATERIAL SPECIFICATIONS	
AS NOTED	

NO.	DATE	DESCRIPTION	REVISION

 Environment Canada Canada Centre for Inland Waters	Environment Canada Centre Canadien des Eaux Interieures
TITLE: A.P.L.L.E. MK. III, MOD. A & B SPRAY BAR	
MOD. A: 36-216 B MOD. B: 36-396/3	
DESIGNED BY: <i>APB</i>	CHECKED BY:
DRAWN BY: <i>K.A.A.</i>	CHECKED BY:
SCALE: <i>N.T.S.</i>	DATE: <i>84-01-19</i>
	APPROVED BY:
	DRAWING NO. <i>ME-5184-5</i>
	SHEET OF

PARTS LIST	
PART NO.	DESCRIPTION



App. I -8

Checked by Checked by Checked by Checked by	Approved by Approved by Approved by Approved by
TITLE APLE 'K III MOD. B PLUMBING FRONT VIEW	DRAWING NO. ME-5984-8 SHEET 2 OF 3

DATE	BY	DESCRIPTION

APPENDIX II

PARTS LIST

Item No.	Part No.	Qty.	Description	Supplier
1		1	55 U.S. gal. stainless steel drum, tight-head 16 gauge, 3/4" & 2" openings, crevice weld, no robar	U.S. Steel of Canada 7 King St. East Toronto, Ont. M5C 1A8
2	K-7083-25	1	All stainless steel magnetic drive, 1/3 hp TEFC electric motor	Cole-Palmer Inst. Co. 7425 N. Oak Park Ave. Chicago, Ill 60648
3	MS-STR-8	1	Swagelok teflon tape	Niagara Valve & Fittings Ltd. 174 Parkdale Ave.N. Hamilton, Ont. L8M 5X2 (Swagelok, Cajon, Nupro, Witey fittings)
4	SS-16-RB-12	1	Reducing bush	
5	B-45S12	1	Ball valve (for MKIIIB Replace with SS4558)	
6	SS-1210-1-12	1	Weld connector	
7	SS-1610-1-16MPW	1	Weld connector	
8	SS-1210-1-12	1	Male connector elbow	
9	SS-810-7-12	1	Female connector	
10	SS-4-BT	1	Branch tee	
11	SS-810-9	4	Elbow (Qty. 5 for MK IIIB)	
12	SS-1210-9	1	Elbow	
13	SS-1210-3	1	Tee (For MKIIIB replace with SS-1210-9 Elbow)	
14	SS-1610-P	1	Plug	

APPENDIX II
PARTS LIST (continued)

Item No.	Part No.	Qty.	Description	Supplier
15	SS-810-3	3	Tee	
16	SS-810-P	2	Plug	
17	SS-810-C	1	Cap	
18	SS-400-R-8	2	Adapter (for MKIIIB eliminated)	
19	SS-8-TA-1-8MPW	2	Adapter	
20	SS-810-1-8MPW	1	Adapter	
21	SS-6-TSW-7-4	1	Connector	
22	SS-YC1-1	2	Check valve	
23	SS-4354	1	Ball valve (for MKIIIB eliminated)	
24	T-813-1	2	Front ferrule	
25	T-814-1	2	Rear ferrule	
26		3ft	1/2" O.D. borosilicate glass tube	any laboratory supply house
27	-009	2	Kalrey O rings (check valves)	Crane Packing Co. Ltd. 423 Green Road Stoney Creek, Ont. P.O. Box 3248, Postal Station C Hamilton, Ont. L8A 7L3
29		3ft	1/4" O.D. x .035" w, type 316 S.S. tube	Atlas Alloys Ltd. 161 The West Mall Etobicoke, Ont.
30		7ft	1/2" O.D. x .035" w, type 316 S.S. tube	
31		3ft	3/4" O.D. x .049" w type 316 S.S. tube	

APPENDIX II
PARTS LIST (continued)

Item No.	Part No.	Qty.	Description	Supplier
32		20ft	3" x 1-1/2" x 1/4" 6063-T6 Alum. Channel	
33		20ft	1" shed 40, 6063-T6 Alum. Pipe	
34		25ft	16 ga. 3 wire cabtire	Rondar Services 333 Centennial Pkwy N. Hamilton, Ont. L8E 2X6
35		1	15 am. U ground plug	
36		1	1/3 HP Motor starting switch	
37		3	Waterproof Feedthroughs C/W metal seals 1/2" NPT	
38		1	Watertight box for starter above	
40	MD-8350- PN	2	Pneumatic swivel casters C/w brakes	Canadian Sling & Cable 1040 Martingrove Rd Unit 21 Rexdale, Ont. M9W 4W4
41	H2R-8250- PN-B	2	pneumatic fixed casters c/w brakes	
42			1/8 6061-T6 Alum. sheet	Atlas Alloys
43		8	1/2"N.C x 2-1/2" S.S. bolts	R.E. Glover Ltd.
44		8	1/2" N.C. x 1" S.S. bolts	
45		32	1/2" flat S.S. washers	
46		16	1/2" S.S. lock washers	
47		16	1/2" N.C. S.S. nuts	

APPENDIX II
PARTS LIST (continued)

Item No.	Part No.	Qty.	Description	Supplier
48		15'	3/4" S.S. Band-it strapping	S.B. Simpson 3210 Mainway Burlington, Ont.
49		2	3/4" S.S. Band-it buckles	
50			16 ga. S.S. sheet	Atlas Alloys
51			1/8" 5056 welding wire	Industrial Welding 520 Beach Road Hamilton, Ont.
52			1/16" 304 E.L.C. welding wire	

APPENDIX III

Cleaning Instructions for A.P.L.L.E. Sampler

1. Disassemble the unit completely, including the valves and the wet end of the pump. Discard the Viton® "O" ring in the pump and replace with a Teflon® unit. Discard the Viton® "O" rings in the safety valves and replace with the Kalrez® ones.
2. Wash all parts thoroughly in hot soapy water, rinse in hot distilled water and allow to dry.
3. Using clean, disposable, gloves, wash all parts in acetonitrile, distilled in glass, followed by an acetone wash, and finally a wash in hexane.
4. Plug all ends of tubes and fittings and lubricate all external threads with Teflon® spray having an isopropanol carrier and nitrogen pressurization and allow to air dry.
5. Reassemble using gloves and washed tools. Use acetonitrile washed Teflon® pipe tape on all tapered pipe threads, starting one thread from the end, and applying two turns of tape.
6. Pressurize to 35 kPa (5 psi) with filtered compressed air or nitrogen and using Snoop® check for leaks at all joints. Tighten as necessary if required. Increase pressure to 105 kPa (15.5 psi) and set the safety valves.
7. Depressurize, and add 5 l of UHP methylene chloride. Circulate for one hour and drain. Concentrate sample as per normal lab procedure in rotary evaporator and check that the chromatograph gives a blank.
8. Cap all openings, to keep out dust and insects, and wrap the funnel and other items, which may come in contact with sample, in degreased aluminum foil.
9. Use extreme care not to contaminate the internals of the system and run blanks of dichloromethane at regular intervals.

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