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## DEFENSE WASTE PROCESSING FACILITY PROTOTYPIC ANALYTICAL LABORATORY

by

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The Defense Waste Processing Technology (DWPT) Analytical Laboratory is a relatively new laboratory facility at the Savannah River Site (SRS). It is a non-regulated, non-radioactive laboratory whose mission is to support research and development (R&D) and waste treatment operations at TNX by providing analytical and experimental services in a way that is safe, efficient, and produces quality results in a timely manner so that R&D personnel can provide quality technical data and operations personnel can efficiently operate waste treatment facilities.

It consists of a 3200 sq ft building that is subdivided into 6 modules comprising 2400 sq ft, Figures 1-6. The remaining 800 sq ft is used for storage, electrical & mechanical equipment, and water still. Each has a sink, appropriate gas lines, bench tops, and cabinets. The modules are sample receiving, chromatography I, chromatography II, wet chemistry and carbon, sample preparation, and spectroscopy. Each module is about 400 sq ft. Most instrumentation is of the same make and model as that at the Defense Waste Processing Facility (DWPF). Also, this laboratory, amongst other work, routinely performs methods that have been designated for use at DWPF. Thus, this laboratory is functionally prototypic of that which is located at DWPF, except for the fact that it is and will remain non-radioactive. The building is also networked to the site's broadband for communication to customers.

For personnel, there are 12 technician analysts, 1 first-line supervisor, 2 B.S. chemists, 2 PhD chemists, 1 programmer, and 1 manager. The laboratory completes, on average, about 150 samples per week for some 1500 results. These samples and analytical work are tracked using the Perkin Elmer Nelson Analytical Laboratory Information Management System (LIMS). The target for sample turn-around is 1 week.

The **chromatography I** module contains organic determination instrumentation. Included here are 3 Hewlett Packard (HP) Gas Chromatographs, HP5890s, with integrators and two of the units are equipped with purge and trap accessories for low level benzene determinations. The lowest reportable value for benzene is <0.5 ppb. Also, there are 2 HP1090 High Pressure Liquid Chromatographs with integrators. Currently, this module reports 13 organics, aniline, phenol, diphenylamine, o-terphenyl, m-terphenyl, p-terphenyl, biphenyl, phenylboronic acid, nitrobenzene, nitrosobenzene, 4-phenylphenol, 2-phenylphenol, and n-phenylformamide with lowest reportable

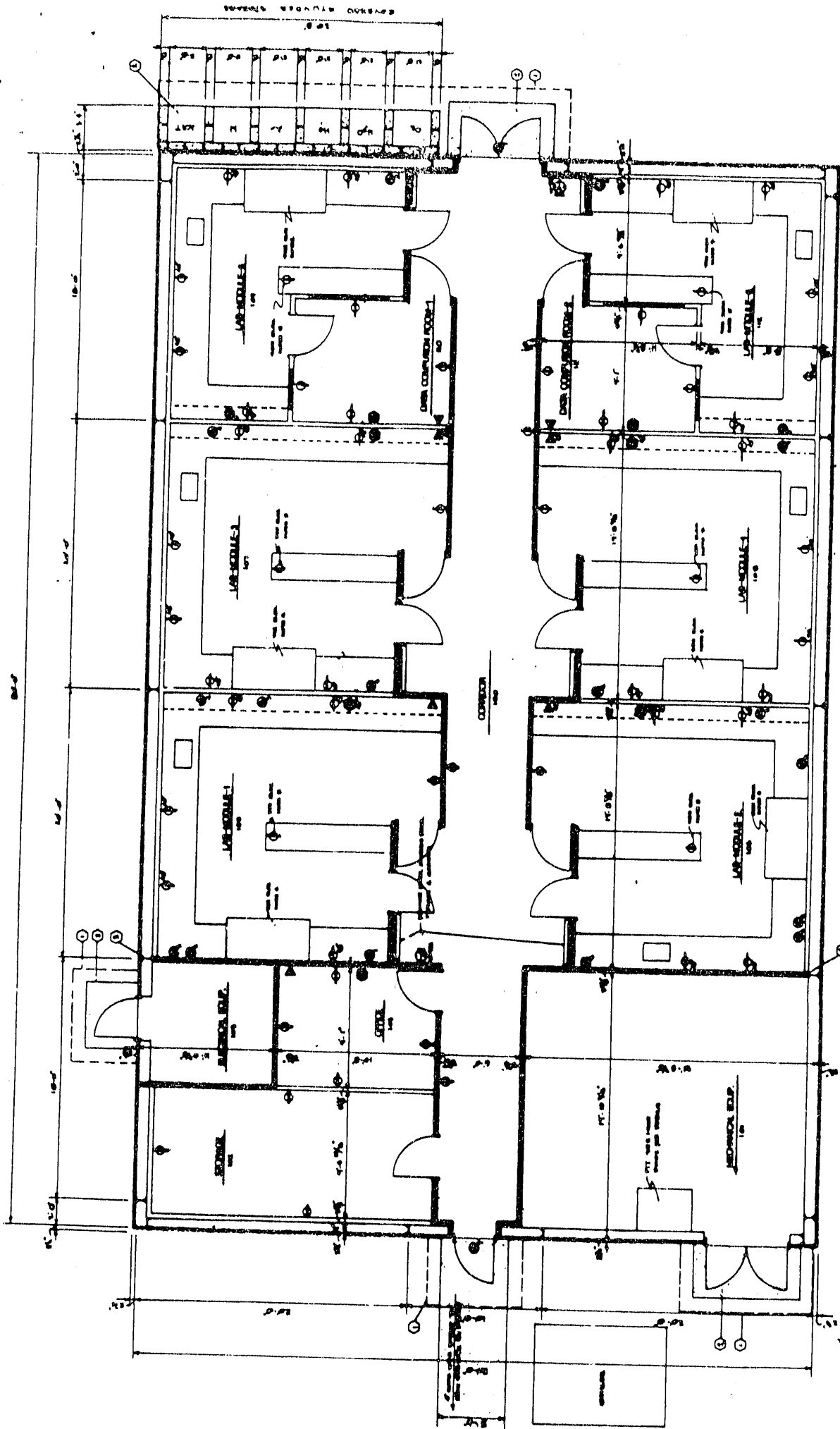
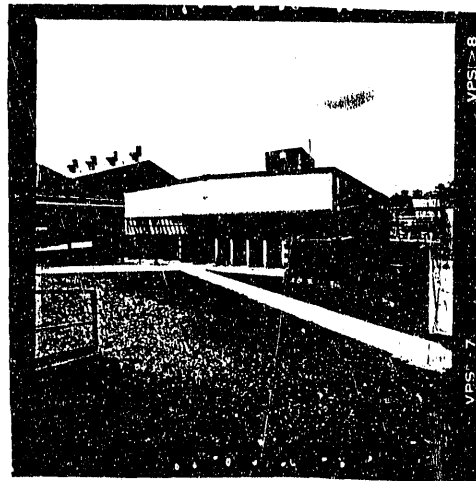
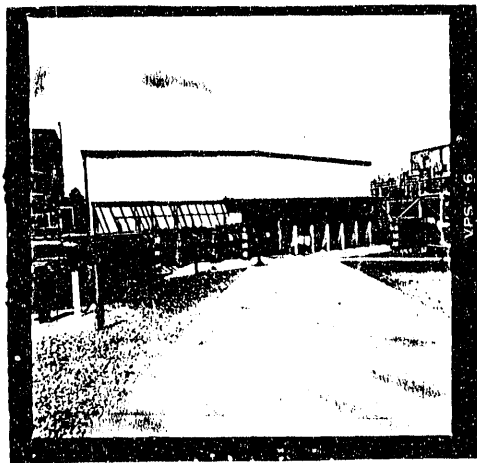


Figure 1

FLOOR PLAN BUILDING 772-T



Figures 2-8 will be photographs of building 772-T, inside and outside, and instrumentation.



*Figures 2A and 2B: DWAT Analytical Lab*

values of <1 ppm. This module has 1 fume hood for sample dilutions, etc. with the organic matrices.

The **chromatography II** module is predominantly ion chromatography (IC). This module has a Dionex 4500i fully automated, dual channel IC and a manually operated Dionex 2100i. The determinations made include the following 8 anions, Fluoride, Chloride, Formate, Nitrite, Nitrate, Sulfate, Oxalate, and Phosphate, and the Ammonium cation. Statistical process control charting indicates that these measurements are all made within 5% error, except for Phosphate which is within 10%. Also, located here is a diode array spectrophotometer, HP8451A, which is used for making colorimetric determinations of hydroxylamine nitrate and iron (II)/iron (III) ratios. This module has 1 fume hood for sample dilution and making reagents.

The **spectroscopy** module houses a sequential Applied Research Laboratories, Inc. (ARL) inductively coupled plasma-atomic emission spectrometer (ICP-AES) Model 3520B and 2 atomic absorption (AA) spectrophotometers, a Varian AA-10 and a Varian AA-400 with 1 graphite accessory. The AA-10 is dedicated for low level mercury (Hg) determinations using Varian's cold vapor accessory (VGA-76). The lowest reportable value for Hg is <0.5 ppb. This module contains 1 fume hood dedicated to the ICP-AES.

The **wet chemistry and carbon** module contains 3 autotitrators for performing various acid/base and/or absolute/equivalent point determinations, 1 microwave oven for measuring solids, pH meters, muffle furnaces for calcining, and 2 OI Analytical Corp. Model 700 Carbon Analyzers. These analyzers measure total organic carbon, total inorganic carbon, and total carbon with lower reportable values of <1 ppm. There is 1 fume hood in this module for the muffle furnaces and sample dilution/measurement of organic containing samples.

The **sample preparation** module contains drying ovens, muffle furnaces, and microwave ovens. Here, heated acid digestions/dissolutions, fusions, and filtering is performed. There is 1 fume hood to house and/or evacuate the ovens and furnaces.

The **sample receiving** module is subdivided into sample storage and an office type area. Samples are logged in and tracked from this point. Results are also stored for a maximum of 1 year, after which the results and their original data are returned to the respective customer. For records, all data is recorded into registered laboratory notebooks and the LIMS.

The LIMS resides on a microVAX 3500 and is a record of all data generated by this laboratory. Upon sample completion, results are automatically sent to customer's mail account, control rooms, and/or printers. The LIMS also generates technician worksheets for the analysts to use.

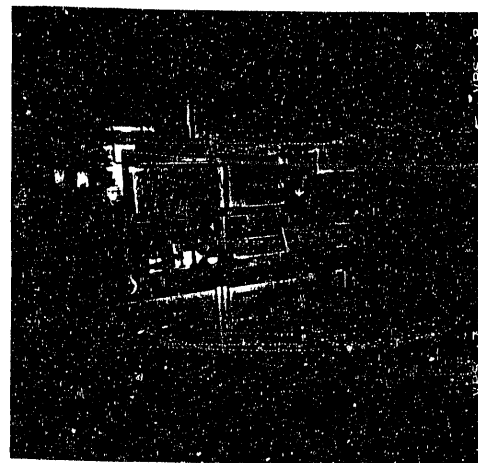
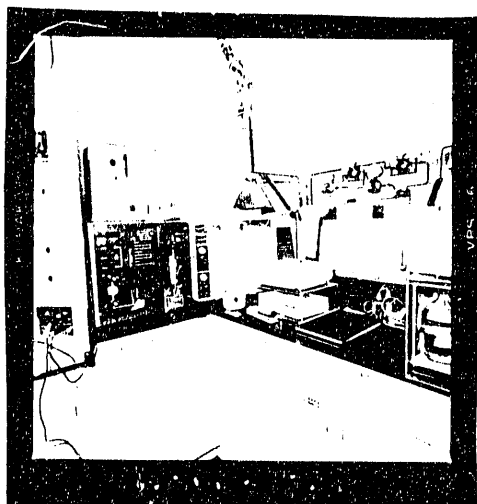
The total cost of this building was 1M of general plant project money. This included the structure, electrical, plumbing, phones, hoods, benches, ICP-AES, AA-400, 4500i, and one GC. The other equipment was purchased separately.

This laboratory supports the following DWPT processes, Integrated DWPF Melter System (IDMS) and Precipitate Hydrolysis Evaporator Facility (PHEF); the following operations processes, Organic Removal Facility (ORF), Ion Exchange (IX), and Effluent Treatment Plant (ETP); 6 individual bench scale researchers located in the area from Interim Waste Technology (IWT), Actinide Technology Section (ATS), and DWPT; and waste analyses for DWPF.

## Chromatography I

organic analyses: benzene (ppm), benzene (ppb), aniline, phenol, biphenyl, diphenylamine, o-terphenyl, m-terphenyl, p-terphenyl, nitrobenzene, nitrosobenzene, 4-phenylphenol, 2-phenylphenol, n-phenylformamide, phenylboronic acid

<u>Instrumentation</u>	<u>Quantity</u>	<u>Notes</u>
HP5890	1	ppm levels of benzene
HP5890/purge&trap	2	detection limit <0.5 ppb
HP1090	2	13 organics detection limit <1ppm



Figures 3A, 3B

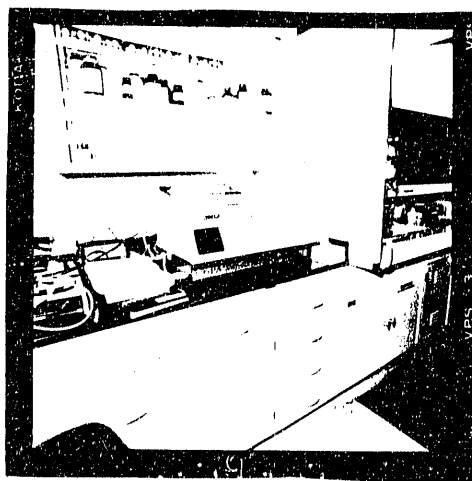
## Chromatography II

ion chromatography: anions --  $F^-$ ,  $Cl^-$ ,  $COOH^-$ ,  $NO_2^-$ ,  $NO_3^-$ ,  $H_2C_2O_4^-$ ,  $PO_4^{3-}$ ;

cations:  $NH_4^+$

colorimetric: hydroxylamine nitrate and iron (II)/iron(III) ratio

<u>Instrumentation</u>	<u>Quantity</u>	<u>Notes</u>
Dionex 4500i	1	detection limit <1 ppm, dual channel, dual autosampler
Dionex 2110i	1	detection limit <1 ppm, single channel
HP8451A	1	



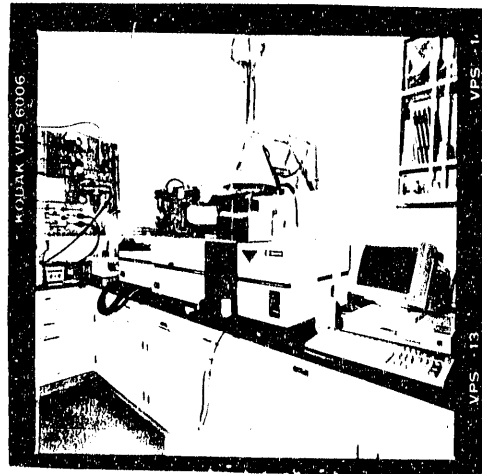
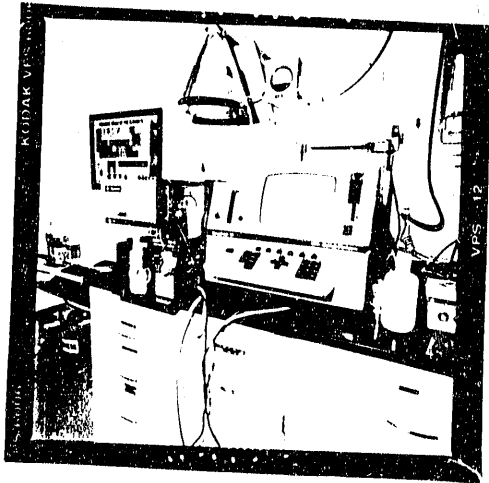
Figures 4A, 4B, 4C



Spectroscopy

Elementals by AA and ICP-AES

<u>Instrumentation</u>	<u>Quantity</u>	<u>Notes</u>
ARL 3520B	1	sequential
Varian AA-400	1	graphite furnace accessory, autosampler
Varian AA-10	1	vapor generation assembly

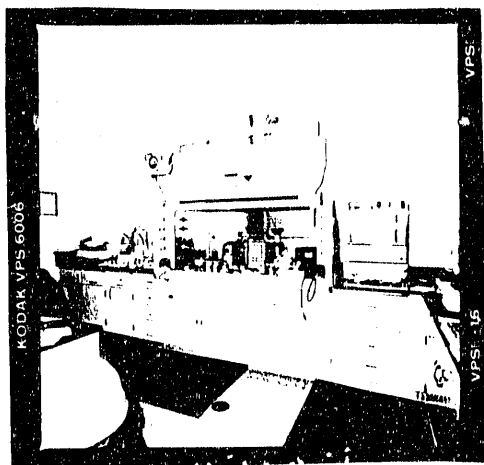


Figures 5A, 5B, 5C

## Wet Chemistry and Carbon

Titration, pH, densities, solids, TOC, TIC, and TC

<u>Instrumentation</u>	<u>Quantity</u>	<u>Notes</u>
OI Corp 700	2	<1 ppm for TOC, TIC, and TC autosampler (2), 1 mL and 10 mL burets
Mettler Autotitrators	3	
pH meters	2	
muffle furnaces	2	



Figures 6A, 6B

## Sample Preparation

Digestions/dissolutions, filtrations

<u>Instrumentation</u>	<u>Quantity</u>	<u>Notes</u>
CEM Model 81	1	Microwave oven, 12 vessel turret
Blue M drying oven	1	95-120°C
muffle furnaces	2	600°C

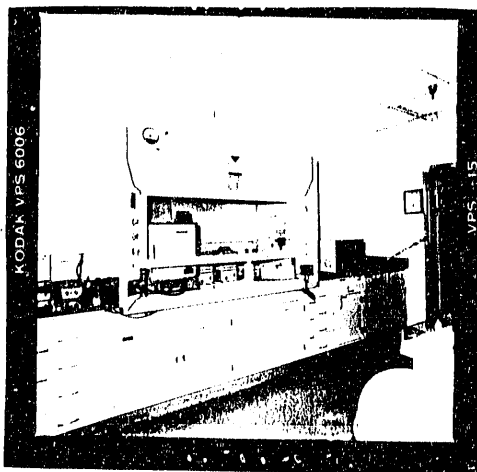


Figure 7

Sample Receiving

Logging, tracking

Instrumentation

Quantity

Notes

PE ACCESS\*LIMS

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networked to site's broadband



Figure 8

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