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MASTER

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SUBJECT: Eurochemic Assistance: Analytical

TO: E. M. Shank

FROM: L. T. Corbin

ACKNOWLEDGMENT

The attached comments were given by personnel of the Analytical Chemistry Division, ORNL, to questions directed to them for the Eurochemic Assistance Program. The original questions, listed in ORNL CF-58-9-91, are included as Appendix I. Rev. 1 has been retyped to facilitate general distribution.

DISCLAIMER

The information requested (Appendix I) is attached. We feel that this information is not complete, and we can give you more detailed information as we become more familiar with this project and have more detailed data from you. The information given herein is relative to the analysis of samples from only uranium-aluminum and uranium-zirconium systems. Since the uranium-stainless steel system is still in the developmental stage, methods for this system have not yet been perfected.

The Oak Ridge National Laboratory Master Analytical Manual can be purchased from the Office of Technical Services (OTS), Dept. of Commerce, Washington 25, D. C. Section 9, "Process Methods," of the Manual has not been printed by the OTS, and they cannot yet give us a publication date. Until it is available, we shall be glad to provide this section.

The Analytical Chemistry Division personnel who may be contacted on specific problems are:

L. T. Corbin	--Process Control
P. F. Thomason	--Ionic Analytical Procedures
S. A. Reynolds	--Radiochemical Procedures
C. Feldman	--Spectrographic Procedures
M. T. Kelly and D. J. Fisher	--Remote Equipment and Instrumentation

They should be contacted by letter, a copy of which we would like to receive.

A large percentage of the instruments and equipment were either developed or modified here at ORNL. Complete sets of drawings and specifications are available. There is a complete description of each instrument and piece of equipment that was developed at ORNL in the ORNL Master Analytical Manual.

Chemical Requirements

1. The following analytical procedures are used routinely:

	Title	Number
a.	Uranium, Spectrophotometric Ammonium Thiocyanate Method	9 052205
b.	Free Acid in Purex Process Solutions	9 052200
c.	Density (Specific Gravity), Fall-Drop Method	9 00600
d.	Total Plutonium, Thenoyltrifluoracetone Extraction Method	9 008621
e.	Tributyl Phosphate in Tributyl Phosphate-Amsco Solutions for Purex Process, Dielectric Constant Method	9 052101
f.	Uranium, Fluormetric Method	9 0071924O

g.	Uranium, Automatic Potentiometric Ferric Sulfate Method	9 0071922h
h.	Plutonium, Potentiometric Ceric Sulfate Method	9 0h32620
i.	Plutonium, Lanthanum Fluoride Method	9 008620
j.	Gross Alpha	9 0733000
k.	Gross Beta Activity in Aqueous or Organic Solutions	9 052302
l.	Gross Gamma, Scintillation Counting	9 0733002
m.	Specific Gravity of a Liquid, Westphal Balance Method	9 00601
n.	Ruthenium Activity in Aqueous or Organic Solutions	2 21731
o.	Niobium Activity in Aqueous or Organic Solutions	2 21551
p.	Zirconium Activity in Aqueous or Organic Solutions	2 21981
q.	Trivalent Rare-Earth Activity in Aqueous or Organic Solutions	2 21992
r.	Controlled Potential Coulometric Titration of Uranium	Not Completed

2. The following types of determinations are required:

a. Uranium
b. Plutonium
c. Tributyl Phosphate
d. Radioactivity (alpha, beta, and gamma)
e. Density
f. Free Acid
g. Fission Products.

It would be impossible at this time to estimate the average number of each determination per month that is required for startup operation, etc. During startup and maloperations, etc., 25 to 50% more determinations will be required than are required for routine operations; 10% more will be required for SSNM and criticality controls.

3. The critical aspects associated with each analysis are discussed very thoroughly in each method contained in the ORNL Master Analytical Manual; the Status section of the method is given over to this discussion.

Physical Requirements

1. In order to estimate manpower requirements, we need the following information: a schedule of control sampling which includes the (1) sample codes, (2) sampling frequency, and (3) desired analyses. Also a list of priority samples, etc., would be helpful.

2. The charge list issued by the Analytical Chemistry Division on January 1, 1958, could be used as a guide if the facility and methods used are similar to those used at ORNL. The analysis time for determinations being done in the HRLAF is also included in this list.

3. The time lapse between sample delivery and submission of results will vary greatly depending on the type of sample, that is, whether it is aqueous or organic, radioactive or nonradioactive, etc., and also on the priority to be given to each determination. For example, the time lapse between sample delivery and submission of results for a uranium determination by spectrophotometric method on a hot dissolver solution would be ~1 hour.

4. Suggested space allocations follow:

   a. Counting and instrument room: 24 ft x 36 ft.

   b. Laboratory space for analysis of nonradioactive samples:
      one room 16 ft x 12 ft for storage of samples
      six laboratories 24 ft x 24 ft.
      This includes room for high-alpha, emission-spectrographic, and radiochemical work.

   c. Laboratory space for analysis of radioactive samples:
      one room 44 ft x 48 ft.
      This includes three high-level cells and one storage cell.

5. The following analytical instruments would be required:

   a. Alpha Proportional Counters
   b. Beta Counters
   c. Gamma Scintillation Counters
   d. Multichannel Gamma Scintillation Spectrometer
   e. Survey Meters and Probes
   f. Spectrophotometers, Beckman Models DU and B
   g. Alpha-Energy Analyzer
   h. Dielectric Constant Meter and Cells
   i. Balances
   j. pH Meters
   k. Emission Spectrograph and Accessories
   l. Falling-Drop Densimeter
   m. Velocity-Servo Potentiometric Titrator
   n. Electronic Controlled-Potential Coulometric Titrator
   o. Filter Photometer
   p. Beckman Model K Titrators
   q. Polarographs
   r. Argonne Model 8 Master-Slave Manipulators
   s. Calculators
   t. Fluorophotometer
   u. Flame Spectrophotometer

APPENDIX I

Information for the use of the Eurochemic Company is desired on the analytical requirements for a combination control and development laboratory which is to be associated with a Purex-type solvent extraction processing plant. This information is to be made available to the Eurochemic Company through E. L. Nicholson. The scope of the information needed, as outlined below, is somewhat tentative and will be modified later as required. It was assumed in preparing the outline that the ORNL Master Analytical Manual is currently available to Eurochemic personnel.

Chemical Requirements

1. What analytical procedures are routinely used?

2. What type of analyses and the average number of each per month are required for startup operation, routine operation, decontamination, and periods of maloperation? (This should include analyses for SSNM and criticality controls, as well as process control.)

3. What are the critical aspects associated with each analysis; i.e., are certain reagents sensitive or unstable and are interfering ions troublesome?

Physical Requirements

1. What are the manpower requirements?

2. How does one apply the charge list as issued by the Analytical Chemistry Division; i.e., what increased time is required per analysis for high-radiation-level samples?

3. What is a realistic time lapse between sample delivery and submission of results?

4. How much space should be allocated for a counting room, for nonradioactive analyses, and for radioactive analyses?

5. What analytical instruments are required?

6. What instruments give particular trouble and what are the maintenance problems?

In addition to the above information, we would like a list of Analytical Chemistry Division personnel who may be contacted on specific problems, such as radiochemical, spectrographic, or ionic analytical procedures and techniques. The desired contact procedure would also be indicated. Any additional information or suggestions will be appreciated.

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