

SECRET

FINAL REPORT  
FIELD TEST FT-34  
ANNEX D  
APPENDIX D1  
ASSAY OPERATIONS  
AND EVASION  
JANUARY 1969

SECRET

D1-1

217

THIS PAGE INTENTIONALLY LEFT BLANK

APPENDIX D1

ASSAY OPERATIONS AND EVASION

A. GENERAL

This appendix presents information on assay of fissile materials and covers some of the problems encountered during assay operations for FT-34. It also presents comments regarding evasion calls by inspectors.

B. ASSAY IN THE OVERALL DESTRUCTION EXERCISE

1. Rocky Flats

a. Problems. No equipment problems occurred during the assay phase of operations at Rocky Flats.

b. Evasion Calls. Both LIMA and MIKE group inspection teams indicated on their data forms that all sample operations were suspect. This "shotgun" effect was possible because of the nonspecific nature of the data forms which permitted teams to indicate that they believed evasion was being used but did not require them to give specific details. Only when teams had a definite reason to discuss evasion did they provide the necessary reason for the call. In general, the "shotgun" approach invalidated evasion calls since specific reasons for evasion were to be supplied for each suspect sample-operation.

Three valid evasion calls were made by LIMA teams and none were made by MIKE teams. One LIMA team noted that during emission spectroscopy the impurities in the sample plus the plutonium totaled more than 100 percent. The team correctly reasoned that contaminants had been added to the sample or carrier and was credited with a valid evasion call. Another LIMA team had marked the carrier material vials prior to loading of the vials and noted that different (unmarked) vials were used later. The team indicated that for the two vials used, a switch had been made and it was credited with

two valid evasion calls because of the observation which concerned two separate samples. (Correct vials were used for the rest of the procedures after discovery of the switch by the observant team.) None of the inspectors observed the tarnished appearance of mass spectrograph filaments. Newly coated filaments would have been bright instead of tarnished in appearance.

2. Oak Ridge

a. Problems. Some minor problems regarding mixed shipments from Pantex and a late shipment from Rocky Flats occurred but did not affect assay operations. Data form problems occurred because of the differences between Oak Ridge standard laboratory forms and the FT-34 data forms. These problems were resolved in the laboratory by substituting some and adding other parts of the standard forms to the field test forms.

The words "large" and "unexpected" appearing on the test data form and relating to impurities required explanation. It was explained that "large" referred to impurity concentrations likely to affect g.U/g. values significantly, or referred to parts per million impurity that, if added, might reach approximately 1000 ppm concentrations. (Inspectors were told to ignore less than 100-ppm values unless they found more than about 10 of them.) "Unexpected" impurities are those not normally encountered because of the technique used. Examples of "normally" encountered large lines are uranium lines (because of the large U-metal content), silver lines (because of the carrier), and carbon lines (because of the graphite electrodes).

Team LIMA 4 accidentally switched samples 7 and 8 while sampling the billets in the foundry. The same thing apparently happened to LIMA 2 and was caused by improper color coding of the sample bottles. This was first detected in the laboratory during a data review of isotopic results.

The mass spectrometer malfunctioned during the analysis by the LIMA 3 team. LIMA 3 was moved out of

SECRET

D1-4

the mass spectrograph room while the machine was repaired. The malfunction was caused by two factors: (1) dirty contacts on the vibrating reed amplifier necessitated a change of amplifier and panel meter. The new meter had a blue-green colored panel rather than the gray panel of the original meter. The inspectors suspected evasion because of this change. However, the change had nothing to do with evasion. (2) A fine wire in the source had arced over to other wires and necessitated a change of source. Inspectors asked and were told of the source change. Again, this had nothing to do with evasion.

A final test problem occurred because of scheduling. It became necessary to tell inspectors who were scheduled to perform chemical analysis the approximate isotopic composition of their samples before they did isotopic analysis in order for them to be able to weigh out the right amount of dichromate for titration. This was also necessary for the military special assay test but not for the civilian special assay since the contractor team did its isotopic analysis first.

b. Evasion Calls. All teams made evasion calls for all sample operations in "shotgun" fashion just as they did at Rocky Flats. The only evasion used was the ratio method in mass spectrometry and this was limited to those samples which contained top-level enrichment. No team correctly identified the evasion tactic used; therefore, no valid evasion calls were credited to any of the inspection teams.

C. SPECIAL ASSAY EXERCISE - MILITARY TEAMS

1. Problems

a. Alloy Casting Problem. During preparation of the aluminum alloy evasion samples, it was found that 2-percent aluminum would not form a homogeneous solution with uranium. On the seventeenth try, a 1.6-percent aluminum, 98.4-percent uranium alloy button was formed and accepted for test even though it would not give the desired 2-percent evasion.

b. Balance Problems

(1) A nut fastening the pan to the CLOUD GAP Mettler metal balance fell into the pan during the weighing of sample 1 by LIMA b. After the balance was repaired by the Y-12 balance technician, LIMA b was offered a choice of 5-gram standard weights to recalibrate the balance. This balance malfunction had nothing to do with evasion.

(2) LIMA d, sample 2 gave a value of 0.92 g.U/g. (instead of a normal reading of ~0.98-0.99) because of a setting error made by the Mettler balance technician. Titer used was over 6 milliliters. The team was advised to rerun the sample using new metal from sample bottle 2. The balance was reset correctly while LIMA d was repickling the sample. The expected value was achieved on the rerun.

c. Evasion Plan Error

(1) The original evasion plan called for exposure of one evaded sample to indicate that evasion could be detected if it were not properly hidden and if proper analytical techniques were used. The sample was to consist of material designated 1502 W which would indicate approximately 8000 ppm of iron by means of emission spectroscopy. Other similar material, designated 1502 T, was to be used for evasion; it would automatically trigger the quantometer to read only 700-800 ppm of iron. During the special assay test, inspectors found a bright orange precipitate for samples containing 1502 material. This precipitate was observed during chemical and isotopic analyses. Because of this precipitate, evasion detection for two of the samples containing 1502 T was a risk because the samples would show evaded readings of an order of magnitude lower than the sample of 1502 W. This would have given away evasion information and the evasion plan had to be altered. All three samples were made up of 1502 T which would automatically trigger the quantometer to indicate lower iron content. When this decision was made, however, team LIMA already had control of its sample, and the ground rules prohibited tampering with samples. Therefore,

for the LIMA special assay, the quantometer was triggered manually so that evasion could be maintained even though each team had a sample of 1502 W material. The first use of quantometer triggering for 1502 material was not successful. All but about 200 ppm of iron was programmed out of the 1502 material for team LIMA a instead of 700-800 ppm. The programming was corrected for succeeding teams, but the error provided giveaway information for LIMA a.

DOE (b)(3)  
DOE (b)(3)

(2) During isotopic analysis of sample 4, team LIMA d obtained a low value of U-235. This low value was caused either by instrument malfunction or contamination of the preplated filament in use during this sample evasion. The low assay value was blamed on recorder malfunction, and the team was advised to rerun the sample. Evasion was then switched to the ratio method, for continuity, because insufficient time was available to change the source and replace the bad filament.

The rigged filament evasion was also unsuccessful for LIMA b for reasons which have not been determined. It is believed that the operator may have forgotten to switch to the rigged filament.

(3) Team MIKE a was analyzing its samples on the quantometer when it became apparent that control sample 7 had no iron and samples 5, 6, and 8 indicated only 300-350 ppm of iron. Investigation revealed that a sticking relay in the evasion circuitry was causing erroneous iron and boron traces on the record. The condition was repaired before other MIKE teams analyzed samples on the quantometer.

DOE  
(b)(3)

2. Evasion Calls. In addition to the giveaway evasion on emission spectroscopy, LIMA teams correctly identified two other evaded sample-operations. MIKE teams correctly identified only one evaded sample-operation. The MIKE teams made several correct calls of evasion for evaded sample-operations; however, all but one of these were invalidated because of incorrect reasons for evasion.

SECRET

D1-7

"Shotgun" effects and suspicion evasion calls were prevalent during the special assay as well as the assay phases of the overall destruction exercise.

In order for an evasion call to be validated, the team had to make a call during an evaded sample operation and give correct reasons for detecting the evasion. Hunches, suspicions, and guesses were not counted as valid evasion calls.

D. SPECIAL ASSAY EXERCISE - CONTRACTOR TEAM

1. Problems

a. Mass Spectroscopy. The contractor team mass spectroscopy expert asked for a rerun of sample 3 (filament evasion) because he felt that the heater current came up too slowly for this sample compared with the other samples. This could have caused a correct evasion call but it was not considered enough justification for such a call by the contractor assay specialist (CAS). On the rerun, the U-235 peak was checked for foreign signals by turning off the peak and checking for a residual signal. None appeared since this was the rigged filament evasion sample. This technique could possibly have uncovered the ratio method of evasion (sample 2), however.

b. Emission Spectroscopy. The contractor team emission spectroscopy expert expressed his desire to pull out the slit and examine it. The request was denied under the ground rule which stated that host equipment was to be used as is. However, no evasion was attempted using the Jarrell-Ash slit. The contractor team expert also wished to weigh the uranium going into the muffle furnace and the oxide coming out, in order to determine whether any volatile material was lost in the process. This request was denied because it did not follow procedures. Actually, weighing would have exposed the rigged balance evasion since oxide would not normally be weighed on the metal balance, and not enough time or money was available to rig every balance available. The contractor emission spectroscopy expert further requested permission to arc an iron spectrum on

the bottom of the spectrographic plate. This request was allowed since it is accepted laboratory procedure in some cases. He deliberately interrupted the arc, thereby effectively identifying the plate to eliminate a "preshot plate" evasion possibility. On the quantometer, the emission spectroscopy expert slipped in a "1000X" (high impurity) standard to check whether every channel on the quantometer was reading. This is a deviation from the ground rule which specified a "20X" (low impurity) standard but it had no effect on evasion since the quantometer would only evade automatically when it sensed the presence of the "trigger" material.

c. Chemical Analysis. The expert chemical analyst carefully washed all flasks, beakers, and entrainment traps with distilled water. He also refused to use the Jones Reductor provided and instead made up his own reductor from a new mercury amalgam bottle. On sample 3, the chemist added a large excess of potassium dichromate over standard procedure and as a consequence had to use a large volume of ferrous ammonium sulphate during titration (about 12 ml instead of 3-½ ml). This could have resulted in low accuracy for this sample. The chemist was extremely suspicious of titration end point on samples 4 and 7. The potentiometer needle oscillated before the end point on these samples only.

d. General. The problems for the contractor team were not those of equipment, apparatus, or design malfunction, but rather were those intentionally created by the team members themselves in their attempt to expose evasion. As noted, the experts used nonstandard methods in their attempts to uncover evasion methods. Some of these methods were disapproved because the tactic was not permitted to military inspectors under the ground rules established for the assay. In one or two instances, the experts carried out their methods without requesting permission. For these cases, the action used did not reveal evasion methods.

2. Evasion Calls. The contractor assay team was unsuccessful in calling evasion. The chemist made two

SECRET

evasion calls on the basis of the strange behavior of the potentiometer needle on two samples. However, these samples were unevaded control samples. Had a "shotgun" method of evasion calls been used, five correct calls would have resulted, although correct reasons may have been lacking. The chemist suspected that the analytical balance had been altered but could not detect any alteration even though he examined the balance closely.

The mass spectroscopist examined the instrument and its electronic components. He was suspicious of the extra wiring but, because of his unfamiliarity with the particular machine used in the test, could not definitely locate or discern any features which he could specifically designate as evasion-producing. Had he continued to increase filament current to burn-out while being evaded by the hidden filament method, he would certainly have discovered the evasion.

The emission spectroscopist did not trust the quantometer and suspected evasion in the use of this instrument but could not detect specifics. His counterevasion tactics on the Jarrell-Ash spectrograph proved ineffective, although some of the actions he wanted to use but was refused permission would have revealed some of the evasion methods.

The contractor inspection team exhibited ingenuity in its attempts to determine evasion methods used. The team was near detection in several instances. The team members were reluctant to call evasion without adequate proof of the method used and did not wish to call evasion for reasons of suspicion. No giveaway information was afforded the contractor assay team.

SECRET

D1-10

226