App. XI

HOT LAB TEST PROCEDURES

CAUTION NOTE

PLEASE NOTE THAT THESE PROCEDURES INVOLVE HANDLING THE UNSEALED SOURCE WHICH CONTAINS 15 MILLICURIES OF RADIOACTIVE MATERIAL NICKEL 63. THESE TESTS MUST THEREFORE BE PERFORMED IN A RESTRICTED AREA (HOT LAB)(EXCEPT FOR SHAKE) AND PERFORMED BY QUALIFIED CHEMISTS AND/OR TRAINED TECHNICIANS USING LAB COATS,FUME HOODS,GLOVES,SAFETY GLASSES,ETC. WHERE APPROPRIATE. NEVER LOOK CLOSELY AT THE RADIOACTIVE MATERIAL WITHOUT SAFETY GLASSES OR OTHER EYE PROTECTION. ALSO KEEP OPEN END OF SOURCE 6" OR MORE AWAY FROM HANDS AND BODY.

1. GENERAL

THE PURPOSE OF THIS DOCUMENT SHALL BE TO TEST NICKEL 63 ELECTRON CAPTURE DETECTORS FOR ALL GAS CHROMATOGRAPHS AND OTHER EQUIPTMENT. THE TESTS ARE LISTED IN TWO DIFFERENT PROCEDURES.

A. TEST ROUTINELY PERFORMED UPON RECEIPT OF INCOMING PARTS.IN SOME CASES ONLY A PERCENTAGE OF EACH BATCH RECEIVED WILL BE TESTED.

B. TESTS PERFORMED TO EITHER QUALIFY A NEW VENDOR OR REQUALIFY AN ACCEPTABLE VENDOR. REQUALIFICATIONS SHOULD BE PERFORMED AT LEAST EVERY TWO YEARS-OR MORE OFTEN IF REASON EXISTS TO BELIEVE A QUALITY PROBLEM MAY EXIST.

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				HOT LAB TEST PROCEDURES	
A	43-10777	J.MARCH	12/03/80	BY JIM PETERS	DATE DEC 3, 1980
LTR	P.C. NO.	APPROVED	DATE	APPD.	SHEET NO. 1 OF 9
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SECTION A

ROUTINE TESTS PERFORMED ON ALL BATCHES OF RADIOACTIVE NI63 PARTS

1. WIPE TEST-SEE FIGURES 1 AND 2.

1.1 THE SURFACE TO BE TESTED SHALL BE WIPED WITH A Q-TIP MOISTENED WITH METHANOL,WIPING AT LEAST 2 SQ. CM. OF OUTER SURFACE.

1.2 THE Q-TIP SHALL BE PLACED IN A NEW SCINTILLATION VIAL (20 ML POLYPROPYLENE)

1.3 10 ML OF SCINTILLATION LIQUID ("BIOFLUOR*" OR EQUIVIALENT) SHALL BE ADDED TO THE VIAL.

*BIOFLUOR-TRADE MARK OF NEW ENGLAND NUCLEAR

1.4 THE VIAL SHALL BE COUNTED IN THE BETA MODE IN A SCINTILLATION COUNTER E.G. BECKMAN MODEL LS100C OR EQUIVALENT.

1.5 THE UNIT IS CALIBRATED WITH A NICKEL 63 STANDARD ALSO CONTAINING A Q-TIP AND SCINTILLATION LIQUID OF THE SAME BATCH AND TYPE AS IN THE TEST MEASUREMENT. THIS CALIBRATION IS PERFORMED WITH EACH BATCH OF MEASUREMENTS MADE.

1.6 THE ACTIVITY OF THE SAMPLE IS CALCULATED AS BELOW:
A=ROOM BACKGROUND (BLANK)
B=COUNTS (PER MINUTE) FOR SAMPLE
C=COUNTS (PER MINUTE) FOR STANDARD
D=ACTIVITY OF STANDARD THAT WAS COUNTED IN MICROCURIES
THEN SAMPLE ACTIVITY =E

$$E = D \times (B-A)/(C-A)$$ IN MICROCURIES

2. VISUAL INSPECTION

2.1 CELLS ARE INSPECTED UNDER A MICROSCOPE* FOR PLATING INTEGRITY TO LOOK FOR CRACKS, FLAKING OR LOOSE PARTICULATES;NONE SHOULD BE VISIBLE.

*MICROSCOPE SHALL BE A STEREO INSTRUMENT WITH VARIABLE POWER OF AT LEAST 10-40.

2.2 PARTICULAR ATTENTION SHOULD BE PAID TO THE CONE END OF THE CAVITY WHERE THE RADIOACTIVE PLATE ENDS. THIS AREA IS MOST LIKELY TO HAVE FLAKING PROBLEMS AND/OR PLATE RESIST RESIDUE.

3. IONIZATION CURRENT

3.1 THE CURRENT IS MEASURED WITH AN ANODE,A .125 DIAMETER METAL ROD WITH A HEMISPHERICAL TIP WHICH PROJECTS INTO THE CAVITY FOR 0.050". THE ANODE IS HELD IN PLACE WITH AN INSULATOR OF 10^12 OHMS RESISTANCE (ANODE TO GROUND) OR HIGHER.

3.2 THE SOURCE IS PLACED ON THE FIXTURE WITH THE ANODE IN THE CENTER OF THE CAVITY.

3.3 A FLOW OF 20 ML/MINUTE OF NITROGEN IS PURGED THROUGH THE CELL CAVITY.

3.4 THE IONIZATION CURRENT IS MEASURED IN NANO AMPS WITH +100 VOLTS DC +/- 10% ON THE ANODE

SECTION B

TESTS PERFORMED ONLY ON QUALIFICATION OF NEW VENDORS OR REQUALIFYING OLD VENDORS.

4. SOLVENT SOAKING
   - 4.1 THE SOURCE SHALL BE PLACED IN A 100 ML BEAKER,FLANGE DOWN.
   - 4.2 25 ML OF LC GRADE METHANOL,10 PPM MAX ACIDITY CALCULATED AS ACETIC ACID (OR OTHER LIQUID AS REQUIRED BY SPECIFICATION) IS POURED GENTLY INTO THE BEAKER AND LEFT FOR 15 MINUTES WITHOUT AGITATION.
   - 4.3 AFTER THE 15 MINUTES HAS ELAPSED THE BEAKER IS GENTLY SWIRLED TO MIX THE LIQUID AND A 1 ML SAMPLE IS PIPETTED INTO A 20 ML SCINTILLATION VIAL.
   - 4.4 10 ML OF SCINTILLATION LIQUID (BIOFLUOR OR EQUIVALENT) IS ADDED.
   - 4.5 THE SAMPLE IS COUNTED AND CALCULATED AS IN 1.4,1.5, AND 1.6
   - 4.6 THE RESULT AS CALCULATED IS MULTIPLIED BY 25 TO GIVE TOTAL NICKEL 63 REMOVED IN SOLVENT WASH. IF OTHER THAN 25 ML WAS USED MULTIPLY BY THE VOLUME USED.
5. HOT WIPE- A WIPE OF THE ACTUAL NICKEL 63 PLATED AREA
   - 5.1 A Q-TIP IS MOISTENED WITH METHANOL AND SHAKEN BRISKLY TO REMOVE EXCESS LIQUID.
   - 5.2 THE Q-TIP IS GRASPED 3-4 INCHES FROM THE COTTON END AND USING A SPIRAL MOTION IS WIPED ACROSS THE PLATED SURFACE WHILE MOVING FROM THE CLOSED TO OPEN END OF THE CAVITY AND MAKING THREE FULL REVOLUTIONS.
   - 5.3 THE PRESSURE ON THE Q-TIP DURING THIS TIME SHALL BE CONSTANT AND LIGHT. IT IS NOT INTENDED TO ABRADE THE SURFACE, MERE TO WIPE ACROSS IT.
   - 5.4 THE Q-TIP IS COUNTED AND CALCULATED AS IN 1.3,1.4,1.5,1.6.
   - 5.5 IF THE COUNTS ARE HIGHER THAN THE EQUIPTMENT CAN ACCOMODATE ,THEN THE SCINTILLATION LIQUID CAN BE DILUTED BY PIPETTING 1 ML INTO A CLEAN SCINTILLATION VIAL AND ADDING AN ADDITIONAL 10 ML OF NEW SINTILLATION LIQUID AN RESULTS CALCULATED AS IN 1.6. SHAKE VIGOROUSLY BEFORE THIS STEP.
   - 5.6 THE RESULTS CALCULATED AS IN 5.5 MUST THEN BE MULTIPLIED BY 10 TO ACCOUNT FOR THIS DILUTION FACTOR. THESE RESULTS ARE APPROXIMATE ONLY.

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6. VIBRATION-ANY SUITABLE COMMERCIAL SHAKE TABLE CAN BE USED

6.1 THE SOURCE SHALL BE MOUNTED INLET DOWNWARD IN A METAL FIXTURE WITH A GLASS FIBER FILTER PAPER BETWEEN THE SOURCE AND FIXTURE -THIS PREVENTS CONTAMINATION OF THE FIXTURE IN THE EVENT OF A PLATING FAILURE.

6.2 THE COLUMN FITTING SHOULD BE SEALED WITH A BLANK PLUG.

6.3 THE SOURCE IS SHAKEN IN ALL THREE MUTUALLY PERPEDICULAR MODES (X,Y,AND Z AXES) BY CYCLING FROM 10-55 HZ FOR A ONE MINUTE CYCLE. THIS IS REPEATED 15 TIMES IN EACH AXIS AT 0.015" PEAK TO PEAK

6.4 USE CARE TO INSURE THAT NO RADIOACTIVE CONTAMINATION OCCURS BY WIPING ALL EQUIPTMENT AND COUNTING BEFORE USE BY ANY OTHER PERSONS.

6.5 AFTER SHAKE TEST, THE FIXTURE IS RETURNED TO THE HOT LAB AND THE CELL REMOVED.

6.6 THE INLET PLUG IS NOW CAREFULLY REMOVED AND BOTH THE PLUG AND THE INLET FITTING WIPED AND COUNTED AS IN 1.1 THROUGH 1.6.

7. DROP TEST-SAME AS A.N.S. N542-1977,TABLE 1.

7.1 A SOURCE IS ASSEMBLED IN FINAL FORM WITH UPPER BLOCK AND THE COLUMN INLET AND GAS OUTLET PLUGGED WITH A STRONG METAL PLUG.

7.2 THE CELL IS DROPPED ("FREE DROP") FROM A HEIGHT OF 1.5M (4.92 FT) ONTO A MILD STEEL PLATE OF 1/4" THICKNESS OR GREATER.

7.3 THIS IS REPEATED FOR TEN DROPS TOTAL. DROP ON DIFFERENT AXIS.

7.4 THE INLET AND OUTLET PLUGS ARE NOW REMOVED AND THE PLUGS AND FITTINGS ARE WIPED AND COUNTED AS IN 1.1 THROUGH 1.6.

7.5 THE DETECTOR SHOULD NOW BE DISMANTLED AND THE ANODE SECTION DISCARDED AS CRACKS IN THE ALUMINA WILL RENDER IT USELESS FOR ANY FURTHER USE.

8. FREEZE TEST

8.1 THE ASSEMBLED CELL IS CAPPED AT INLET AND EXIT FITTINGS WITH METAL CAPS AND PLACED IN A SEALED PLASTIC BAG.

8.2 THIS CELL IN PLASTIC BAG IS NOW PLACED IN A -40 DEG C CHAMBER (A REFRIGERATOR-FREEZER MAY BE SUITABLE OR AN ENVIRONMENTAL CHAMBER) FOR A PERIOD OF 20 MINUTES.

8.3 THE CELL AND BAG ARE NOW TAKEN OUT AND ALLOWED TO REACH ROOM TEMPERATURE (THIS REQUIRES ONE HOUR OR MORE).

8.4 THE CELL IS REMOVED FROM BAG AND THE INLET AND EXIT FITTINGS REMOVED AND WIPED WITH Q-TIPS SOAKED IN

METHANOL. THE BAG IS DISPOSED OF IN RADIATION DISPOSAL CONTAINER.

8.5 THE Q-TIPS ARE COUNTED AS IN 1.1 THROUGH 1.6.

9. HIGH TEMPERATURE NICKEL LOSS- THIS TEST SIMULATES WORST CASE THERMAL RUNAWAY OF ALL TEMPERATURE CONTROL SYSTEMS. PERFORM ALL TESTS IN A HOOD.

9.1 THE DETECTOR SHOULD BE MOUNTED ON A METAL BRACKET AND PLACED IN A HIGH TEMPERATURE (625 DEG C) OVEN WITH THE INLET FITTING OPEN.

9.2 THE EXIT IS ATTACHED TO 1/8" STAINLESS STEEL TUBE WHICH LEADS OUTSIDE THE HEATED ZONE.

9.3 THE 1/8" LINE IS CONNECTED TO A SERIES OF FOUR FRITTED GLASS WASHING BOTTLES (FISHER 3.040 SIZE 125 ML) VIA A SMALL PIECE OF SURGICAL RUBBER TUBE.

9.4 THE CONTENTS OF THE WASH BOTTLES ARE: FIRST EMPTY,SECOND 15% NITRIC ACID (60 ML), THIRD WATER (60 ML) AND LAST 1% ALCOHOLIC DIMETHYL GLYOXIME SOLUTION.

9.5 ALL BOTTLES ARE ARRANGED SO THAT THE FRIT IS ON THE INLET SIDE OF THE BOTTLE.

9.6 A VACUUM PUMP IS CONNECTED TO BOTTLE 4 AND REGULATOR (A SIMPLE VALVED "T"). THIS ALLOWS FLOW OF AIR TO BE REGULATED SO THAT MODERATE BUBBLING OCCURS.

9.7 NOTE THAT AIR IS USED. AS IN ANY HIGH TEMPERATURE TESTS ALL "O"-RINGS, GRAPHITE FERRULES, OR GLASS COLUMNS WILL HAVE DEGENERATED AND WILL HAVE ALLOWED THE SYSTEM TO BE FULL OF AIR DURING MOST OF THE RUNAWAY PROCESS.

9.8 THE CELL TEMPERATURE IS NOW RAISED TO 625 +/- 25 DEG C AND MAINTAINED FOR THE TEST TIME (24 HOURS).

9.9 THE CELL IS NOW COOLED AND THE VACUUM SOURCE DICONNECTED.

9.10 60 ML OF 15% NITRIC ACID IS NOW ADDED TO THE FIRST (EMPTY) BOTTLE AND LEFT FOR 15 MINUTES.

9.11 1 ML OF EACH OF THE FIRST THREE BOTTLES IS PIPETTED INTO A SCINTILLATION VIAL AND NUETRALIZED WITH 1 NORMAL SODIUM HYDROXIDE USING PHENOLPTHALEIN AS AN EXTERNAL INDICATOR. CHECK ALL BOTTLES FOR RADIATION WITH A GEIGER TUBE PROIR TO PIPETTING OUT THE 1 ML.

9.12 THE SAMPLES ARE COUNTED AFTER ADDING 10 ML OF SCINTILLATION LIQUID (BIOFLUOR).

9.13 IF THE LAST BOTTLE TURN PINK OR RED (INDICATING THAT NICKEL HAS REACHED THE LAST BOTTLE) ADDITIONAL CAUTION SHOULD BE USED AS LARGE AMOUNTS OF NICKEL MAY HAVE BEEN RELEASED.

9.14 THE SAMPLES ARE COUNTED AND AND CALCULATED AS IN 1.3 THROUGH 1.6 WITH THE ADDITIONAL STEP OF MULTIPLYING EACH

RESULT BY THE VOLUME OF LIQUID LEFT IN EACH BOTTLE AFTER THE TEST. EVEN THOUGH 60 ML WAS USED TO START, ALL OF THE BOTTLES MAY HAVE EVAPORATED AT DISSIMILAR RATES.

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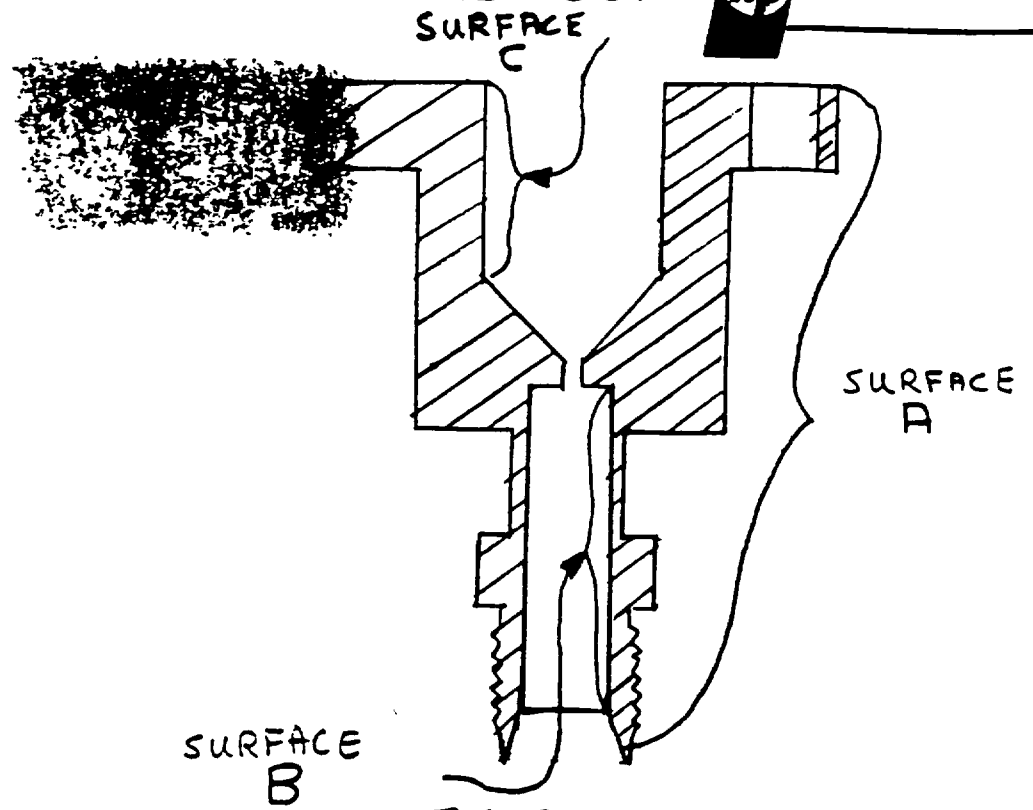

FIGURE 1

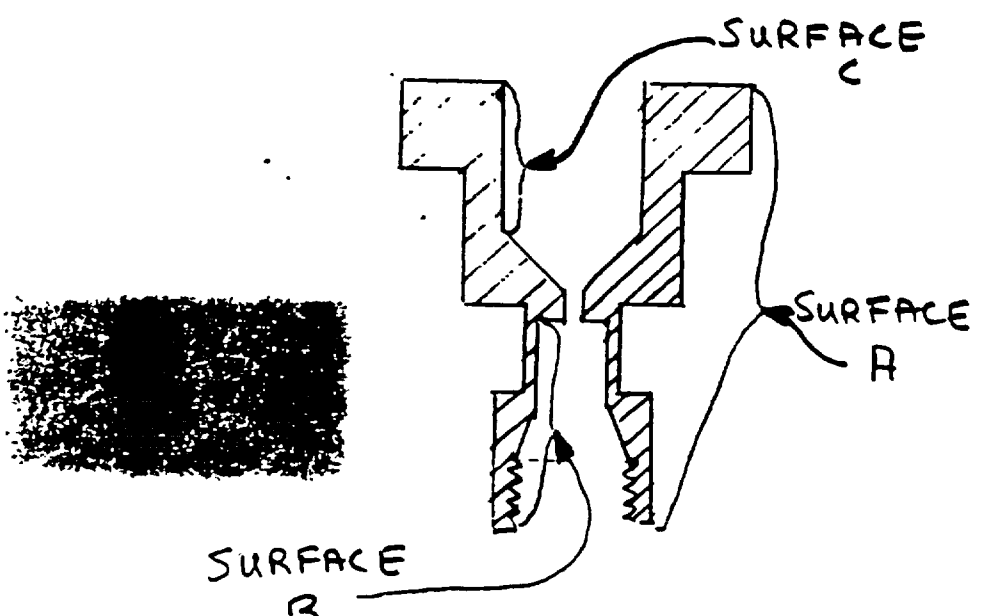

FIGURE 2

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SPECIFICATIONS FOR P/N 19303-

TEST#	TEST NAME	%TESTED	
1	INLET WIPE SURFACE B FIGURE 2	100	2 NANOCURIES MAX AT INCOMING, INPROCESS,AND FINAL INSPECTION
1	BODY WIPE SURFACE A FIGURE 2	100	5 NANOCURIES MAX LEGAL (REPORTING) LIMITS,AT FINAL INSPECTION (AFTER ASSY&GC TEST)
2	VISUAL PLATING INSPECTION	100	NO VISIBLE PEELING,CRACKS OR LOOSE PARTICLES
3	ION CURRENT	100	9(+/-10%) NANOAMPS OR 8.1 TO 9.9 NANOAMPS
4	SOLVENT SOAK	SECTION B 10 SOURCES MINIMUM	LESS THAN 10 NANOCURIES REMOVED IN 15 MINUTE SOAK IN LC GRADE METHANOL 25 ML USED FOR TEST
5	CAVITY WIPE SURFACE C FIGURE 2	AS REQUIRED	5 MICROCURIE MAX
6	VIBRATION (SHAKE)	MINIMUM OF 1	LESS THAN 5 NANOCURIES REMOVED FROM SURFACE A&B (FIGURE 2). ALSO PASS TEST 2 (VISUAL)
7	DROP TEST	MINIMUM OF 1	LESS THAN 5 NANOCURIES REMOVED FROM SURFACE A&B (FIGURE 2) ALSO PASS TEST 2 (VISUAL)
8	FREEZE TEST	MINIMUM OF 1	LESS THAN 5 NANOCURIES REMOVED FROM SURFACE A&B (FIGURE 2) ALSO PASS TEST 2 (VISUAL)
9	HIGH TEMPERATURE RUNAWAY	MINIMUM OF 1	LESS THAN 0.5 MICROCURIES LOST IN CARRIER GAS.

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