

OPERATION MANUAL MODEL KOCOUR 6000 STEP TESTING with (NF6MS) STAND



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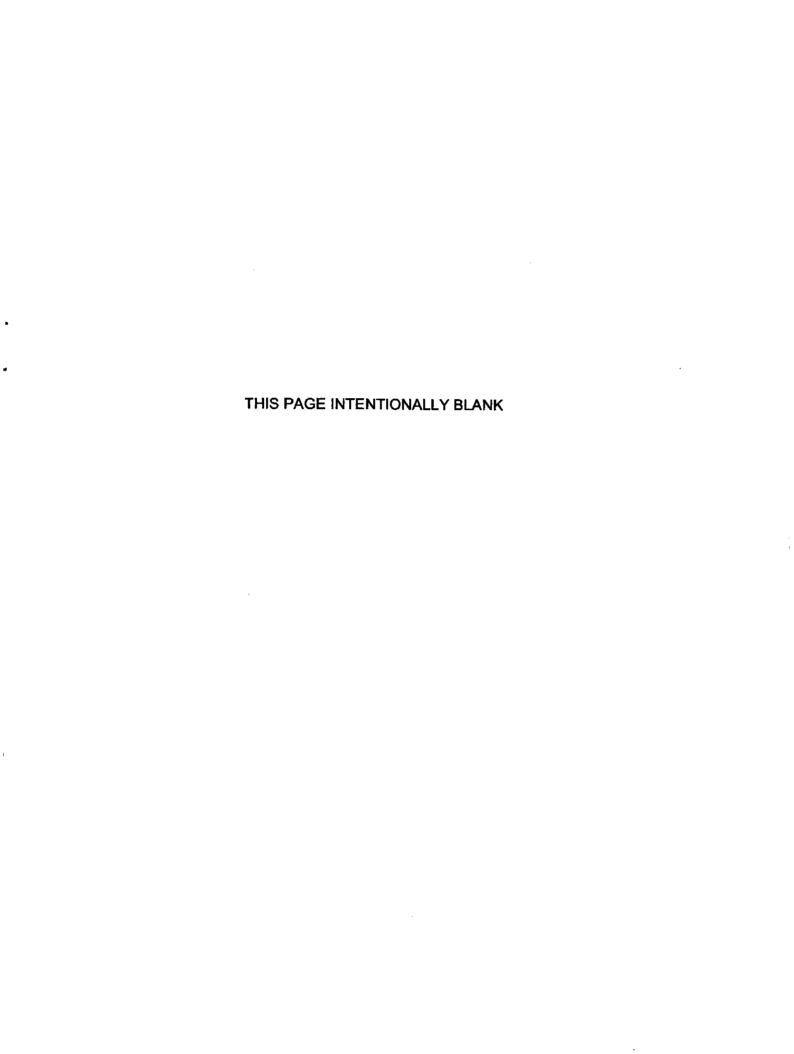


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STAND and ELECTRODE ASSEMBLY INSTRUCTIONS

EQUIPMENT REQUIRED

- 1. Kocour 6000 Thickness Tester
- 2. Step Stand (NF6MS)
- 3. Strip Chart Recorder
- 4. Electrode Kit

CONTENTS OF ELECTRODE KIT

- 1 Electrode assembly (pre-wired) w/black sleeving
- 1 STEP "A" agitator assembly
- 1 Nickel foil
- 1 Stainless "A" cell
- 1 "A" gasket
- 1 Plastic syringe (3 cc) for filling GR tubes
- 1 Vial ("A" reference electrolyte with 2 GR tubes)
- 1 3M KCI SAT'D with AgCI
- 1 60 ml waste solution bottle
- 1 125 ml electrode storage reservoir solution with 2 caps
- 1 Cell cleaning brush
- 1 Instructions

PROCEDURE FOR TEST STAND SET-UP (refer to STEP Stand schematic)

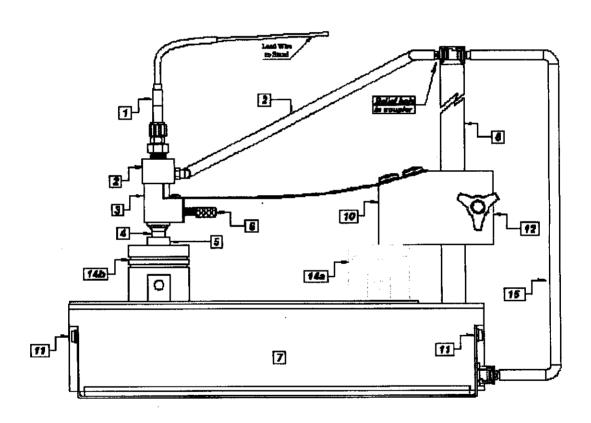
- 1. Read all instructions first.
- 2. Connect black patch cable to the receptacle at the rear of the STEP stand and to the receptacle marked "Accessory" on the front panel of the 6000.
- 3. Insert stainless steel cell (4) into cell holder (3) and lock into position with cell set screw (6).
- 4. Connect the black cable as follows; Insert banana plug into the receptacle in the head of the cell set screw (6). Insert the black pin connector to the black receptacle at the rear of the STEP stand.
- 5. Insert the pin connector on the red cable to the red receptacle in the front of the STEP stand. The alligator clip at the other end of this cable is used to connect the part (14) being tested.
- 6. Connect agitation tubing to the Luer fitting at the rear of the stand (7) and to the fitting on the side of the agitator assembly (2).
- 7. Carefully remove electrode assembly from electrode box. To prepare Step Test electrode, refer to "Assembly, Filling and Operating Instructions" on page 4 for Step Test Electrode (single junction).
- 8. Insert red pin connector of electrode assembly (1) into the red receptacle at the

rear of the stand (7).

- 9. Attach the spade connector on the black wire of the electrode assembly to the negative (-) terminal on the recorder.
- Attach the spade connector on the red wire of the electrode assembly to the 10. positive (+) terminal on the recorder.
- Preparing recorder: Set recorder to: Input Range 0.5 V; Chart Speed 3; and 11. Chart - CM/Min.

Electrode assembly is now prepared for testing.

STEP STAND SCHEMATIC



- STEP-TEST electrode assembly (single junction) 1)
- 3) Cell holder
- Gasket ** 5)
- 7) Measuring stand base
- Height Set Slider Gauge 10)
- Height and Angle adjustment knob 12)
- 17" Port Extension (Optional) 15)

- 2) **Agitator Assembly**
 - Stainless steel cell **
- 4) 6) Cell set-screw
- Vertical support post 8)
- Red and Black Jacks 11)
- Test specimen (by customer) 14)

The "A" cell and "A" gasket must be used for the STEP Procedure. RELIEF HOLE IN COUPLER (as shown above) MUST REMAIN OPEN! NOTE:

STEP TEST ELECTRODE (SINGLE JUNCTION) ASSEMBLY, FILLING AND OPERATING INSTRUCTIONS (MI - 401)

The Micro-Reference Electrode has an internal silver-silver chloride reference electrode using a reference electrolyte of 3 M KCl saturated with silver chloride. This reference electrolyte (A) is provided in the electrode box. The junction is a ceramic frit sealed in a Glass Reference tube (GR tube).

Before the electrode can be used, one GR tube must be removed from the 28ml vial and refilled with the reference electrolyte (A) provided. This is accomplished in the following manner:

- A. The reference electrolyte (A) is added to the GR tube with the syringe assembly.
 - 1. Withdraw 1 ml of reference electrolyte with the syringe.
 - Carefully insert the tubing of syringe assembly into the GR tube until the tip of the tube touches the ceramic frit at the base of the GR tube.
 - Using the syringe, slowly inject the reference electrolyte while removing the small plastic tubing.
 There should be no air bubbles in the GR tube. If air bubbles are present, repeat step 1, 2 and 3.
- Electrode agitation assembly. (refer to electrode agitator assembly sketch on page 5)
 - Unscrew and remove Luer Part #XMTLL-1 from electrode cap.
 - Insert the filled GR tube into the threaded end of Part #XMTLL-1.
 - Insert the silver wire (Ag-AgCl electrode) into the GR tube (The silver wire should not touch the
 junction). Screw Part #XMTLL-1 together with the filled GR tube into the electrode cap (Do not
 over-tighten). After assembly, the bottom of the GR tube must be 1/4"-5/16" from the tip of the
 agitator.
 - Insert GR tube into pre-assembled agitator assembly (3-70-T38).
 - 5. The flow of reference electrolyte through the junction is mandatory for proper operation of the "Step Test". As a result of this flow, the upper level of reference electrolyte in the glass tube will recede approximately 1/16" 1/8" (1.5 3.0mm) per day. This electrolyte must be replenished. Check the electrolyte level in the GR tube daily and refill with reference electrolyte if necessary. (Always check for presence of air bubbles)
 - 6. Should the junction become clogged, soaking in warm (not hot) electrolyte will unclog it. A spare GR tube is enclosed so that you may continue testing while the other tip is soaking. The filled GR tube should be left submerged in electrolyte until it is needed for further testing. Make sure electrode wire is dry before re-inserting into GR tube. Submersing the wet wire into the GR tube will dilute the electrolyte.

STORAGE and CLEANING of ELECTRODE

The Electrode Storage Reservoir is supplied with two caps, a shipping cap and a storage cap (cap with SFTL-6 fitting). Remove the shipping cap and replace it with the storage cap.

SHORT TERM (1 week)

Place the filled GR tube with silver wire still inserted into storage Solution (B) by removing luer fitting (XMTLL-1) from fitting (SFTL-6) with a 1/4 turn counterclockwise, twist and place this assembly thru storage cap into Solution (B) and tighten with 1/4 turn clockwise.

NOTE: Always clean electrode before long term storage

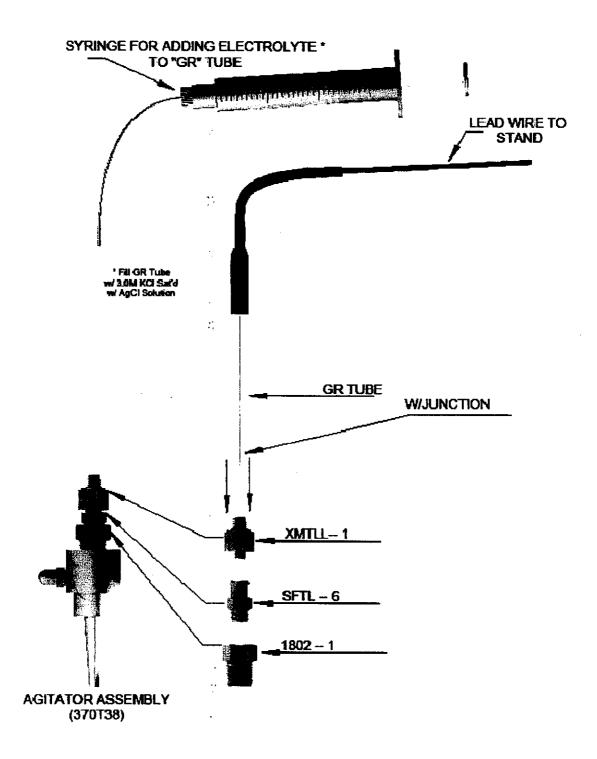
LONG TERM:

Remove the silver wire from the GR tube and rinse with D.I. water and pat dry using an absorbent towel; replace black sleeve and store in original case. Place electrolyte filled GR tube in reference electrolyte (A) and replace cap.

SPECIFICATIONS:

Internal Element	Silver-Silver Chloride (Ag-AgCl electrode)
(A) Reference Electrolyte	3 M KCl sat'd with AgCl
(B) Electrode Storage Reservoir Solution	3.0 M KCl solution

ELECTRODE AGITATOR ASSEMBLY MI - 401 (MOD)



STEP TEST PROCEDURE

COMMENTS

The Step Test is an extremely sensitive electrochemical procedure. Therefore, the cleanliness of the cell is essential. In particular, carefully follow the procedures for filling, rinsing, and storage of the electrode as outlined on page 4. Always use a properly cleaned and <u>nickel coated</u> stainless steel cell. (Refer to Section 2 below)

SECTION 1: POSITION SAMPLE (refer to Measuring Stand lay-out drawing on page 3)

- 1. Set selector to Ni on tester and have "A" Module fully inserted.
- 2. Set the test sample at a position (14) under the Height Set Slider Gauge (10).
- 3. Lower the Height Set Slider Gauge (10) until it touches the test area on the sample. Lock the Height Set Slider Gauge in this position with the Height and Angle adjustment knob (12).
- 4. Carefully lift the Cell Holder (3) and slide the test sample from position (14a) to position (14b) under the gasket (5). Center the gasket on the test area. Steps 1-3 assure that the pressure on the gasket is proper for testing.

SECTION 2: COATING CELL

- 1. Position the Pure Nickel foil into the test position.
- 2. Fill the test 2/3 to 3/4 full with STEP SOLUTION. (Check foil surface for air bubbles)
- 3. Insert the Electrode Agitator Assembly (page 4) with the reference electrode assembly into the Cell Holder (3).
- 4. Attach the red alligator clip to the nickel foil.
- 5. Re-check the settings on the 6000 and the chart recorder.

6000 (front)	RECORDER
Power Switch ON	V/mV selector to V - V range selector to 0.5
Plate Selector Switch L/O	REC - STBY switch to STBY - Chart speed selector to 3
Setting Ni	Power switch "ON" - CM/MIN-OFF-CM/HR to OFF

- 6. To start the test:
 - (A) Move CM/MIN OFF CM/HR selector to CM/MIN position.

(B) Move REC-STBY switch to REC. © Press "GO/NOGO" switch on 6000.

At this point the pen should peg left on the chart paper. Use the zero adjust control knob to move this line to 3cm. from the left margin of the chart paper. The trace line should be smooth and without erratic motion. (If line is not smooth, clean the SS cell with wire brush supplied and ensure bottom of GR tube is at the correct depth in the agitator assembly, repeat Sec. 2).

- 7. Continue this procedure until the 6000 display reads 1.50 mil. Then:
 - (A) Press "GO/NOGO" switch on 6000
 - (B) Move REC-STBY switch to STBY.
 - (C) Move CM/MIN OFF CM/HR switch to OFF.
- 8. The cell is now coated and ready to perform the "STEP" test. This coated cell should now produce from 10 to 20 reliable "STEP" tests. The exact number depends on the nickel thickness on the test samples and the purity of the nickel plate tested. **CAUTION:** <u>DO NOT</u> rinse electrode, agitator assembly, cell or gasket between continuous testing.

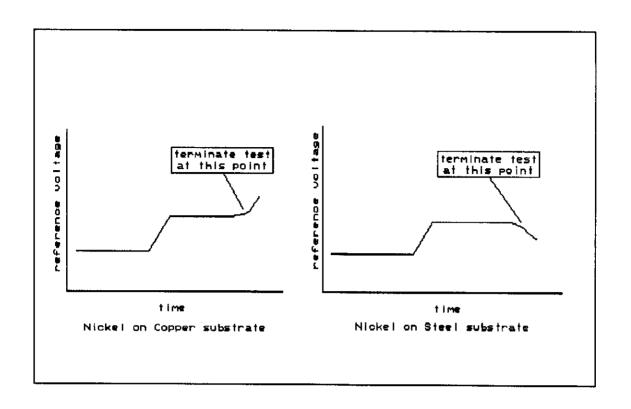
SECTION 3: PERFORMING STEP TEST

- 1. Position the test sample into the test position (14B). **NOTE:** Decorative chromium should be removed before STEP test.
- 2. Proceed with steps 2,3,4 and 6 in previous section.
- Watch the chart trace and manually terminate at the points indicated. (See figure on page 8)
- 5. Do not allow test to continue beyond these points because the copper and iron removed will contaminate the cell.
- 6. Interpreting test (Refer to page 8).

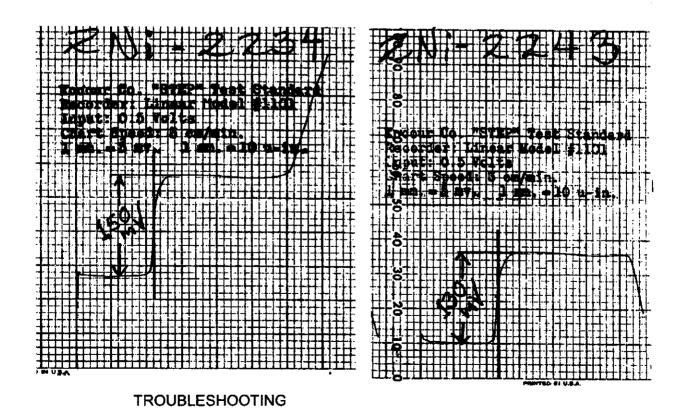
SECTION 4: STRIPPING TEST CELL

- 1. The cell must be stainless steel. The stainless steel cells are typically marked: "SSA"; "SS"; A; "Ā"; or A.
- 2. Rinse with D.I. water and clean with wire brush, rinse again and dry. The cell is then ready for coating.

STEP TRACE ILLUSTRATION



BELOW, ACTUAL TRACINGS OF STEP STANDARD



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SYMPTOM	PROBABLE CAUSE	REMEDY
Erratic pen movement when	Dirty cell	Clean w/brush & water
nickel coating S.S.	Uneven coating	Strip and re-coat
cell	GR tube blocking air flow on agitator	Readjust GR tube to free obstructions (see page 4)
	Air bubbles in GR tube or cell	Check GR tube and cell
	Fluctuating line voltage	Check line voltage
Chart pen pegs off scale and stays left even after moving zero knob when test	GR tube is blocking agitator air flow	Readjust GR tube (see page 4)
is started	Solutions too low or contaminated (ref. electrolyte A or STEP)	Replace
	Ag-AgCl wire damaged	Replace
	Ceramic frit clogged	Unclog(see troubleshooting Sec. B 6 in MI-401 Instr.)
Balance meter deflects between Nickel layers, but chart recorder shows no STEP (see example on page 6)	V-mV selector switch stuck between 1 and 0.5 volts	Reseat voltage range to 0.5v. Retest
Chart pen leaves a triangular trace. Little	Clogged GR tube tip	Change GR tube
or no response		Refer to Section B troubleshooting in MI-401 Operating Instructions
	Electrode wire defective	Replace electrode
Pen has no movement, remains at right index mark	Rec-STBY switch is in STBY position	Set to REC position
at right index mark	Module not plugged in fully	Reseat module
	Poor/no pin connections in stand or recorder	Check all pin connections
	Patch cable loose/not plugged into 6000 or stand	Plug in patch cable