MICROSENSORS FOR in-situ MONITORING OF Cr and U IN GROUNDWATER IN DOE SITES

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GOALS and EM NEEDS

- THIS PROJECT ADDRESSES THE EM NEEDS FOR ON-SITE CHARACTERIZATION OF CONTAMINATED NUCLEAR ENERGY SITES.

- THE RESEARCH WILL CREATE POWERFUL AND ECONOMICAL MICROSENSOR SYSTEMS FOR in-situ MONITORING OF PRIORITY METAL CONTAMINATS (particularly U and Cr) IN DOE FACILITIES.

RATIONALE

CHEMICAL SENSORS, CAPABLE TO OPERATE IN-SITU OR ON-SITE, HOLD A PARTICULAR PROMISE FOR FIELD MONITORING OF METAL CONTAMINANTS.

SUCH DEVICES CAN OFFER IMMEDIATE DETECTION OF SUDDEN METAL DISCHARGE AND RAPID FEEDBACK AND GUIDELINE DURING REMEDIATION ACTIVITIES.

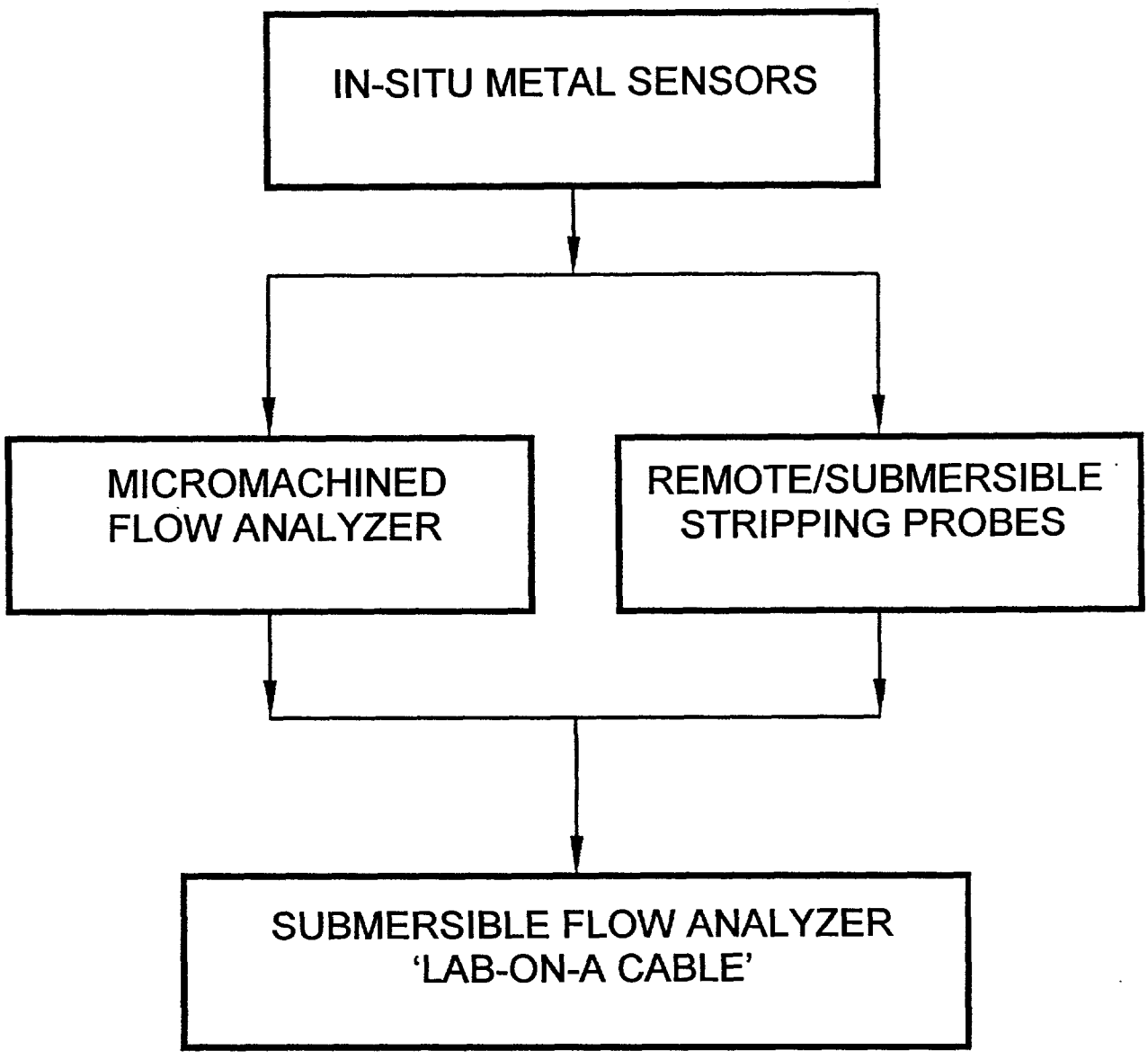
PARTICULARLY ATTRACTIVE ARE NEW SENSORS BASED ON ELECTROCHEMICAL STRIPPING ANALYSIS.

ELECTROCHEMICAL STRIPPING ANALYSIS

ONE OF THE OF THE MOST POWERFUL TECHNIQUES FOR TRACE METAL MEASUREMENTS. IT REMARKABLE SENSITIVITY IS ATTRIBUTED TO ITS "BUILT-IN" PRECONCENTRATION STEP, DURING WHICH THE TARGET METAL IS ACCUMULATED (by deposition or adsorption) ONTO THE ELECTRODE.

IN ADDITION TO ppt DETECTION LIMITS, SA OFFERS THE ADVANTAGES OF MINIATURIZATION

APPROACH


IN-SITU METAL SENSORS
MICROMACHINED FLOW ANALYZER
REMOTE/SUBMERSIBLE STRIPPING PROBES
SUBMERSIBLE FLOW ANALYZER 'LAB-ON-A CABLE'

MICROMACHINED (μTAS) STRIPPING ANALYZER

BENEFITS OF MICROMACHINED STRIPPING ANALYZER

- RAPID AND AUTOMATED SAMPLE PROCESSING
- MINIMAL USE OF REAGENTS AND SAMPLES
- ECONOMY (low cost per device and for sample)
- PORTABILITY (smallest metal analyzer, 1x1x1 inch)
- LOW POWER REQUIREMENTS
- REMARKABLE SENSITIVITY

MICROMACHINED STRIPPING ANALYSIS FLOW SYSTEM

MICROMACHINED SYSTEMS WOULD ALLOW PERFORMING ALL THE STEPS OF THE STRIPPING ASSAY ON MINIATURIZED SILICON ELEMENTS.

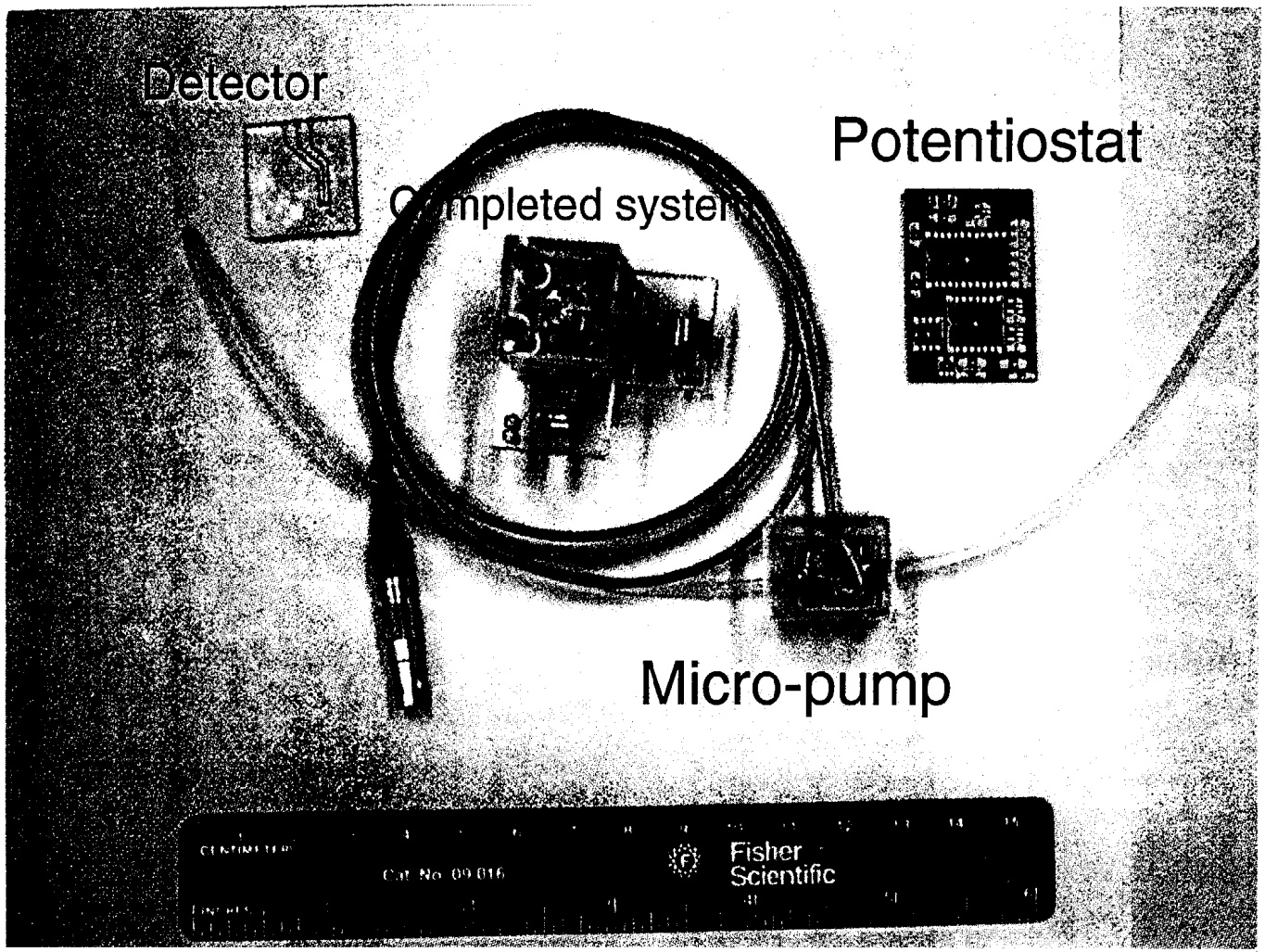
SUCH ON-CHIP OPERATION WOULD ALLOW TESTING FOR TRACE METALS TO BE PERFORMED MORE RAPIDLY AND EASILY IN A FIELD SETTING.

MINIATURIZED STRIPPING FLOW ANALYZER

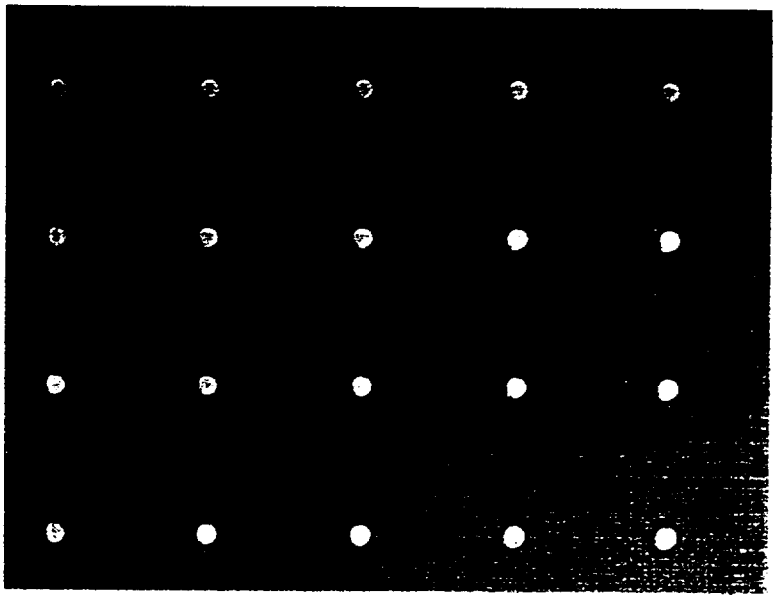
MICROFABRICATED BY MICROMACHINING PATTERNING OF 3D-MICROSTRUCTURES (pumps, channels, etc.).

INTEGRATES ALL THE STEPS OF THE STRIPPING ASSAY (both sample handling and detection), FOR A FASTER AND SIMPLER OPERATION IN A FIELD SETTING.

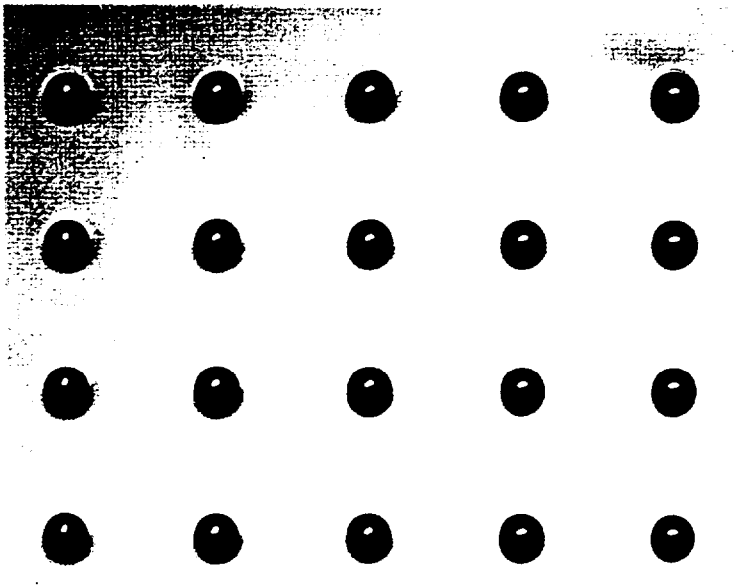
Electrochemical Micro Total Analysis System



Array of Iridium-based Mercury Microelectrodes

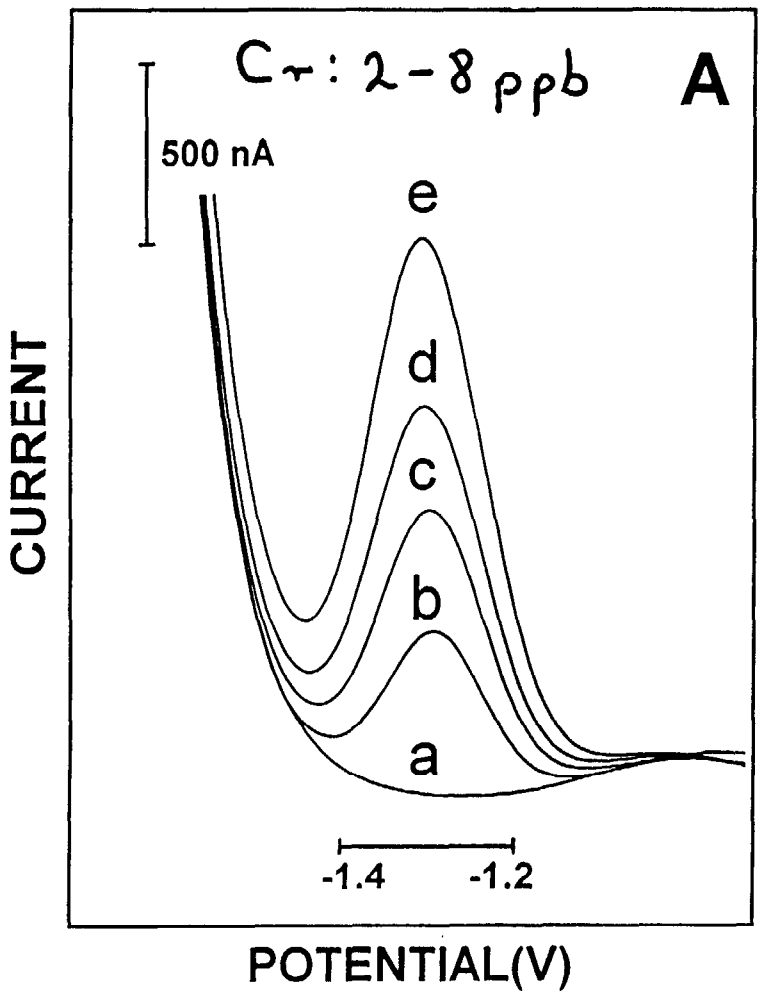


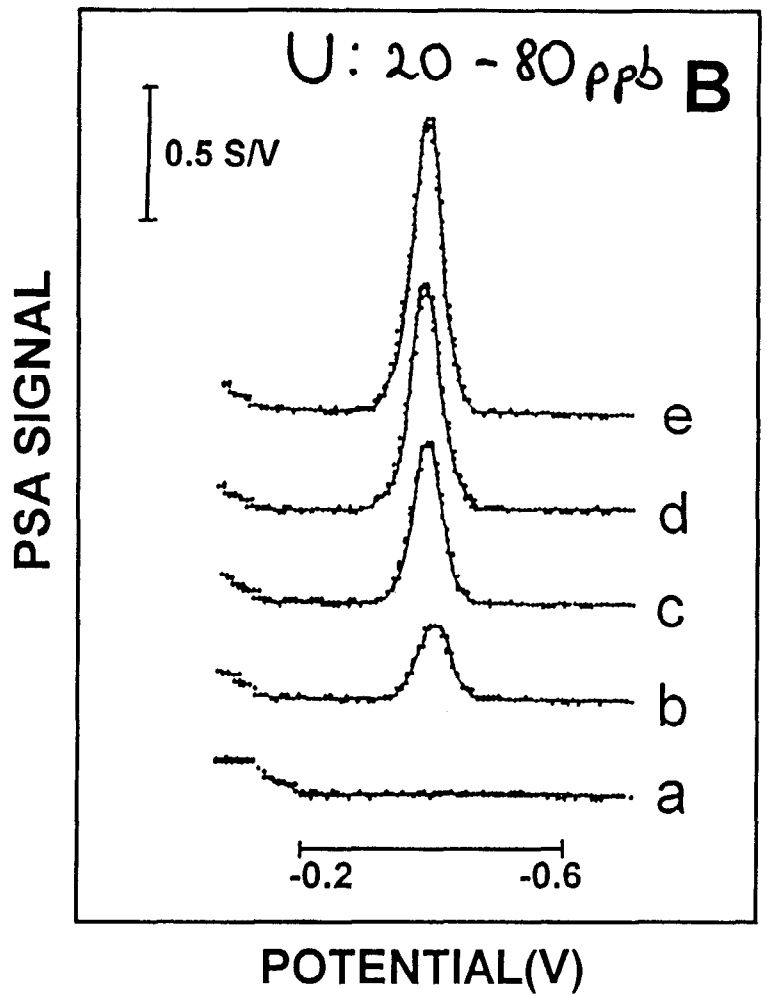
Bare Iridium



Mercury Coated

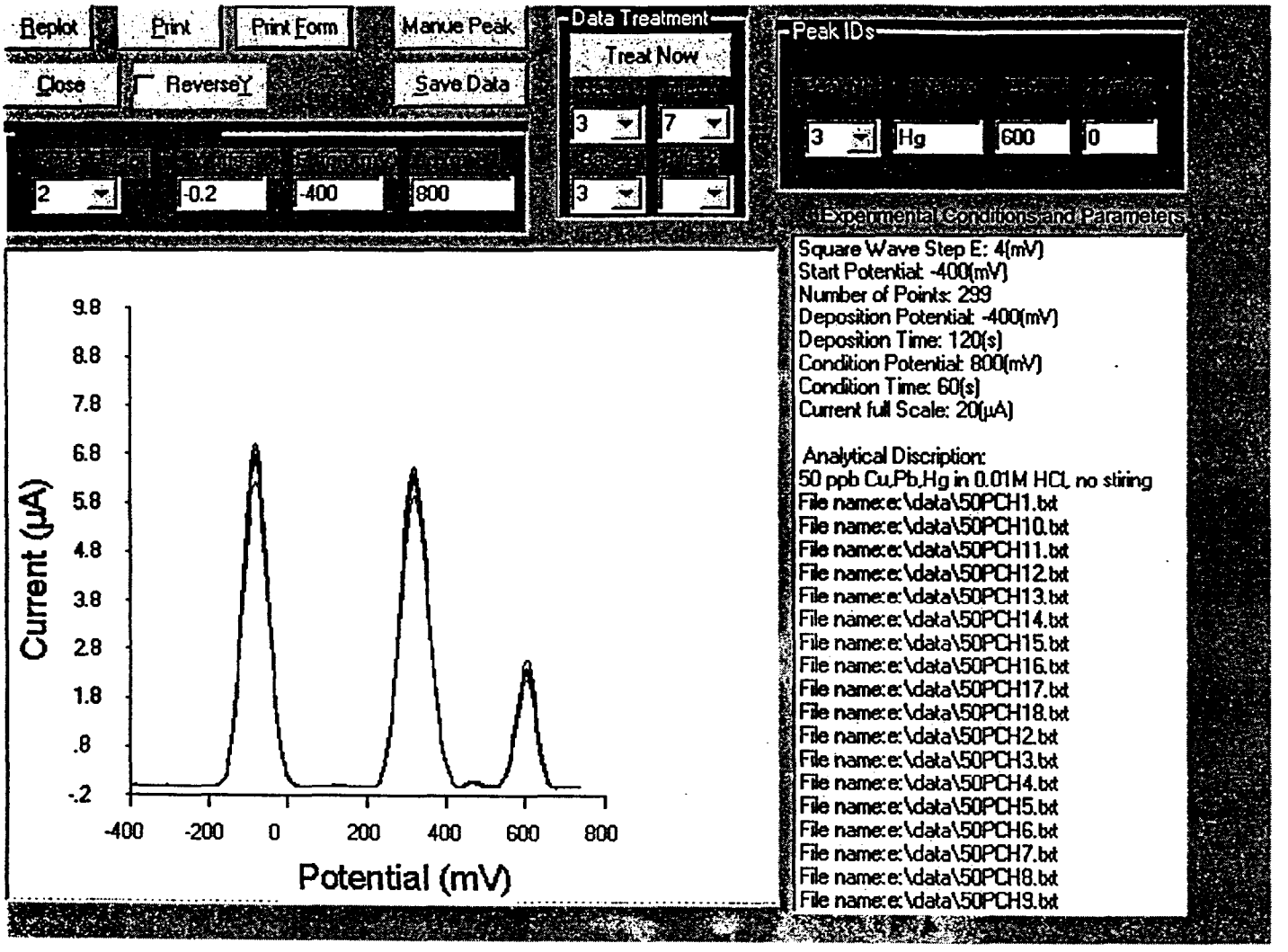
ELECTROCHEMICAL μ-TAS
FOR MONITORING OF TRACE CHROMIUM(VI) and URANIUM (VI)





A: SQUARE WAVE VOLTAMMOGRAMS OF CHROMIUM(VI). a. 0; b. 2; c. 4; d. 6; e. 8ppb of Cr(VI). Adsorption Time, 40s.

B: CHRONOPOTENTIOGRAMS OF URANIUM (VI). a. 0; b. 20; c. 40; d. 60; e. 80ppb of U(VI). Adsorption Time, 60s.

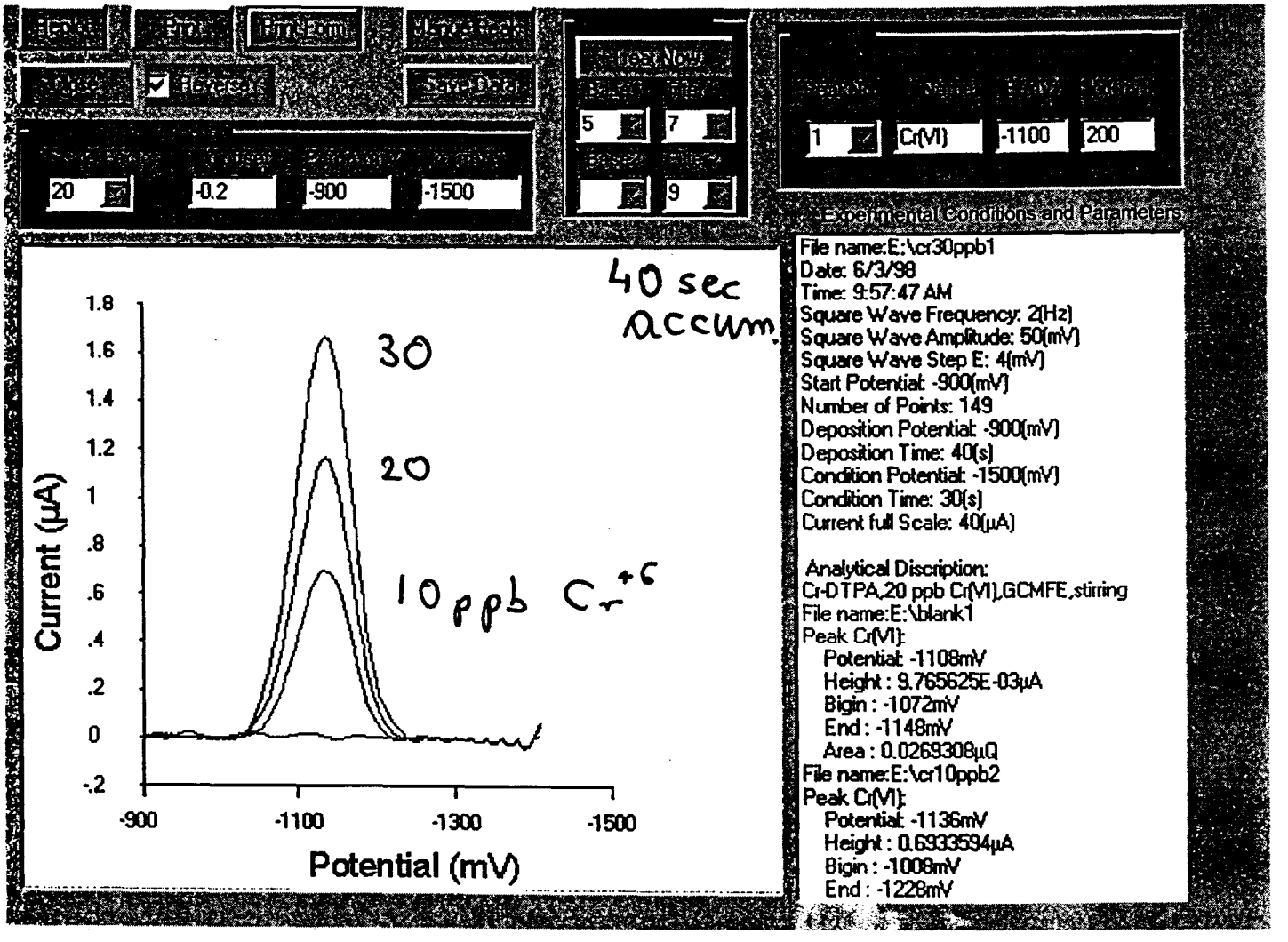


ON-CHIP POTENTIOSTAT – 20 REPETITIVE SWASV MEASUREMENTS OF 50ppb Pb, Cu and Hg (gold fiber sensor; 2 min accumulation)

ON-CHIP NMSU POTENTIOSTAT

MINIATURIZED SQUARE-WAVE VOLTAMMETRIC ANALYZER

STRIPPING MEASUREMENTS OF Cr(VI) – 10ppb INCREMENTS

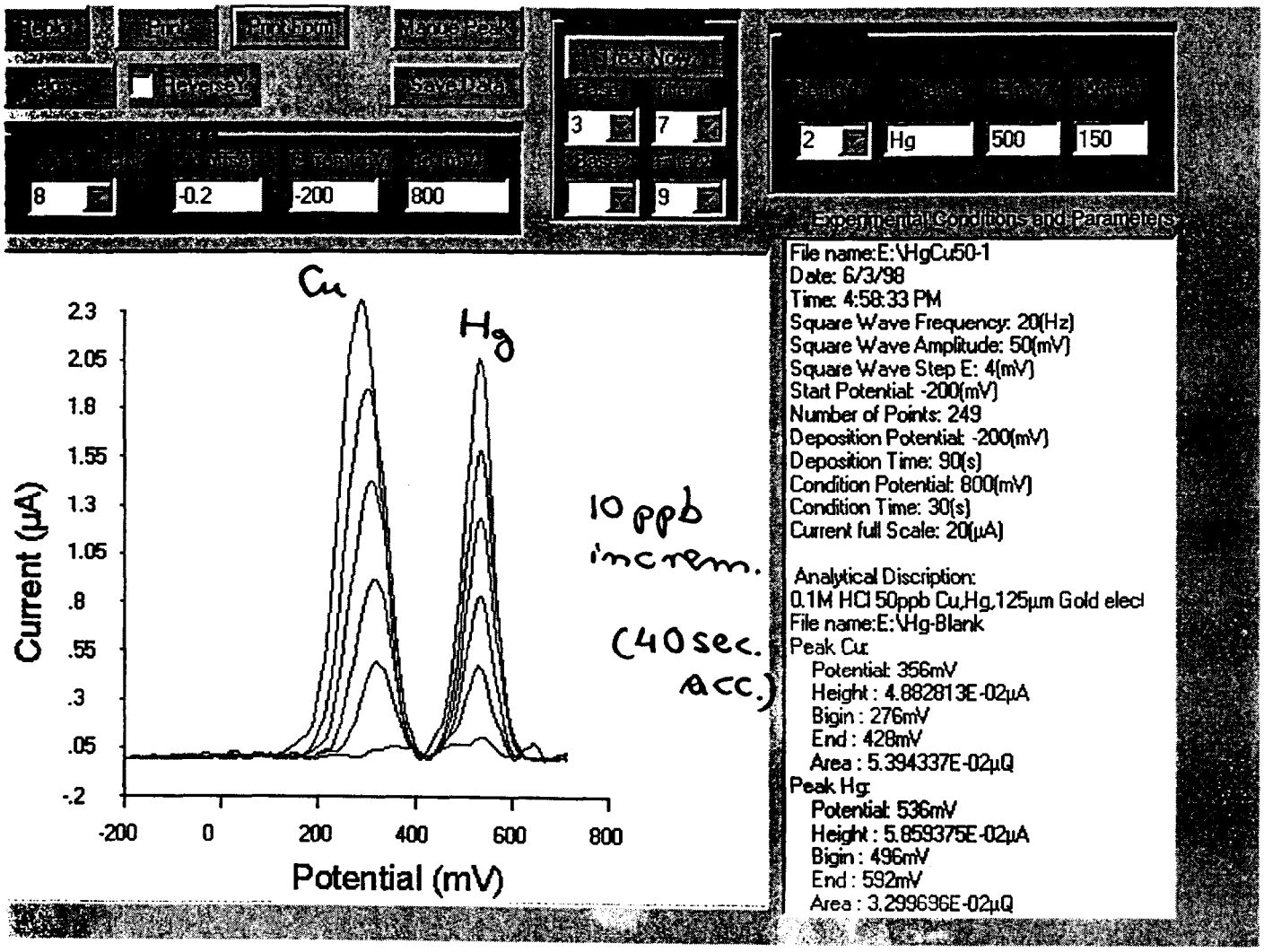


Typical voltamograms recorded with a mini square-wave voltammetric analyzer which is proposed to be embedded to a remote prob. Glassy- carbon mercury film electrode, solution containing 0.01M DTPA, 0.04M acetate buffer, 0.5M sodium nitrate, and with successive additions of 10 ppb Cr(VI) Deposition potential: -0.9V(vs. Ag/AgCl reference), deposition time 40s; square-wave frequency: 2 Hz, amplitude: 50 mV, step potential 4 mV.

ON-CHIP NMSU POTENTIOSTAT

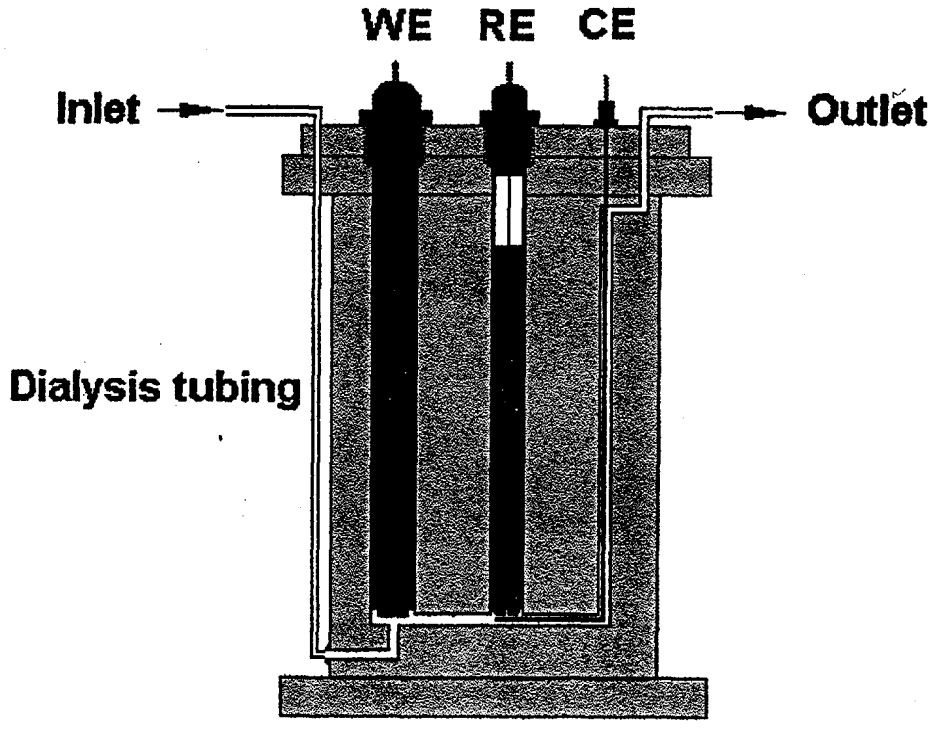
MINIATURIZED SQUARE-WAVE VOLTAMMETRIC ANALYZER

RESPONSE TO 10PPB INCREMENTS IN THE CONC. OF Cu and Hg
at a GOLD WIRE ELECTRODE

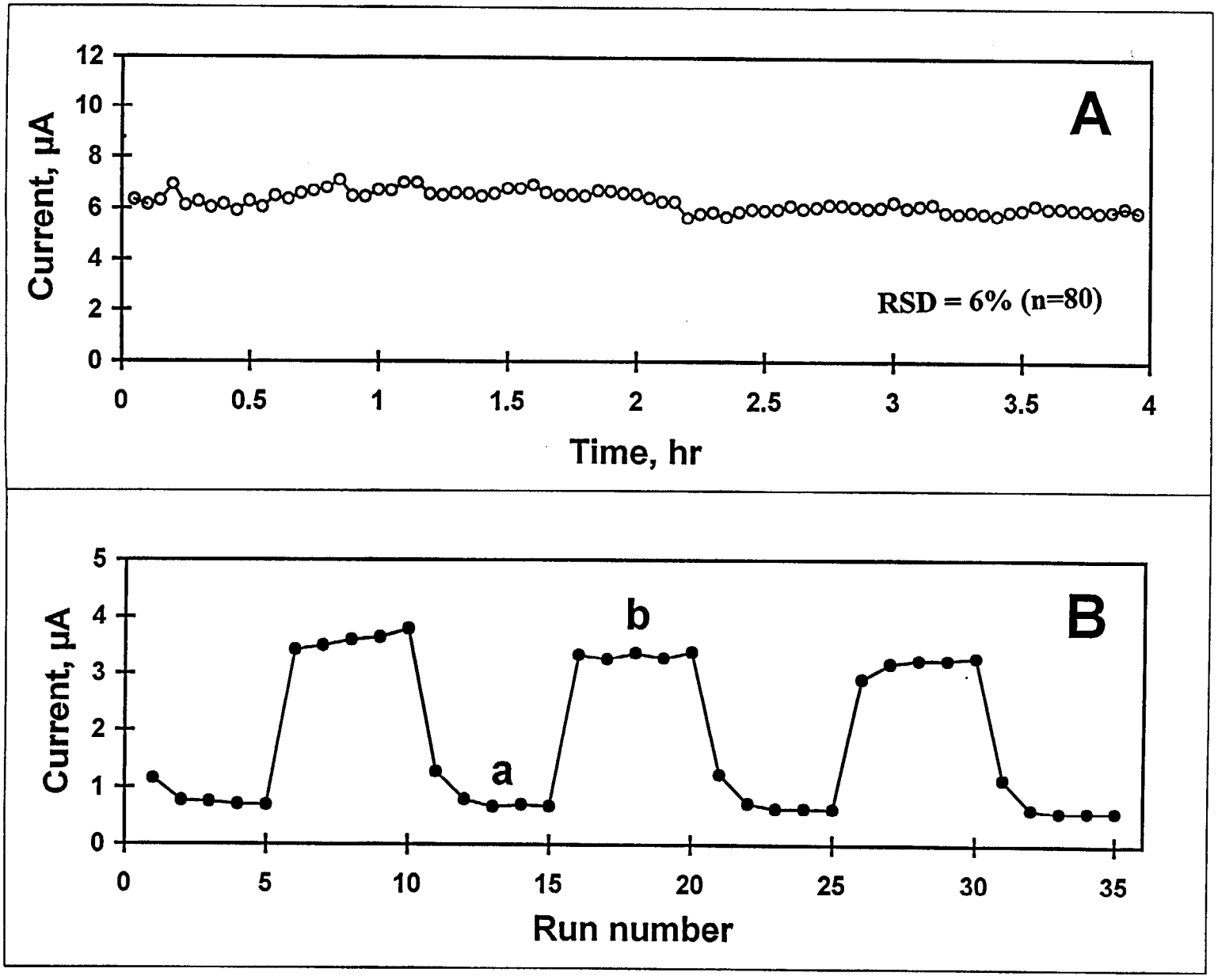


Typical voltamograms recorded with a mini square-wave voltammetric analyzer which is proposed to be embedded to a remote prob. Gold cylindrical electrode(10-mm length, and 0.1-mm diameter); 0.1M HCl with successive additions of 10 ppb Cu(II) and Hg(II); Deposition potential: -0.9 V (vs. Ag/AgCl reference), deposition time 40s; square-wave frequency: 20 Hz, amplitude: 50 mV, step potential 4 mV.

Renewable-Reagent Remote Sensor



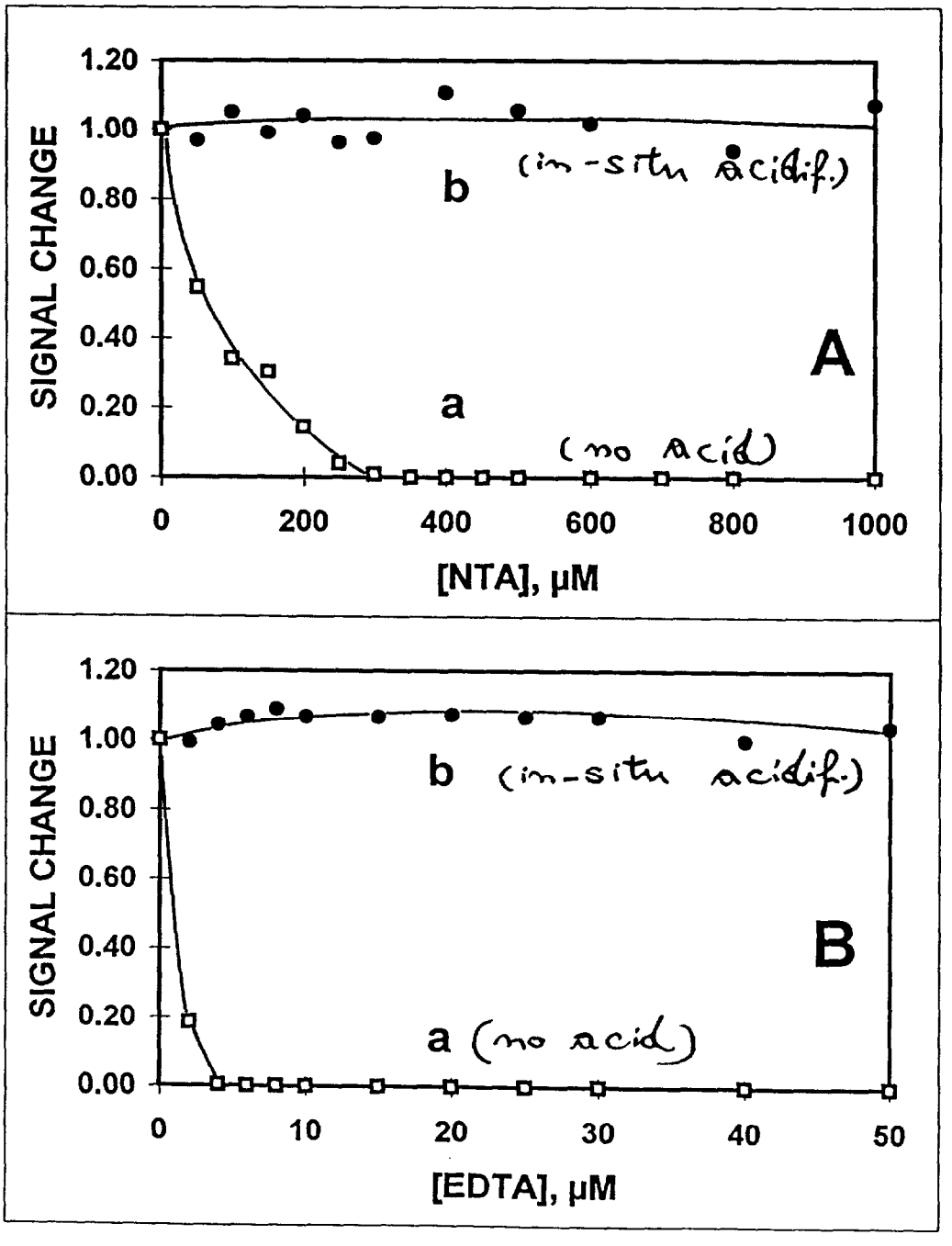
SUBMERSIBLE CHROMIUM SENSOR



STABILITY (at 5 ppb) and CARRY OVER (5⇔25 ppb) EXPERIMENTS

IN-SITU SENSING OF THE TOTAL METAL CONTENT
(based on internal acid delivery)

MONITORING OF Cd and Pb IN PRESENCE OF EDTA AND NTA



Cd

Pb

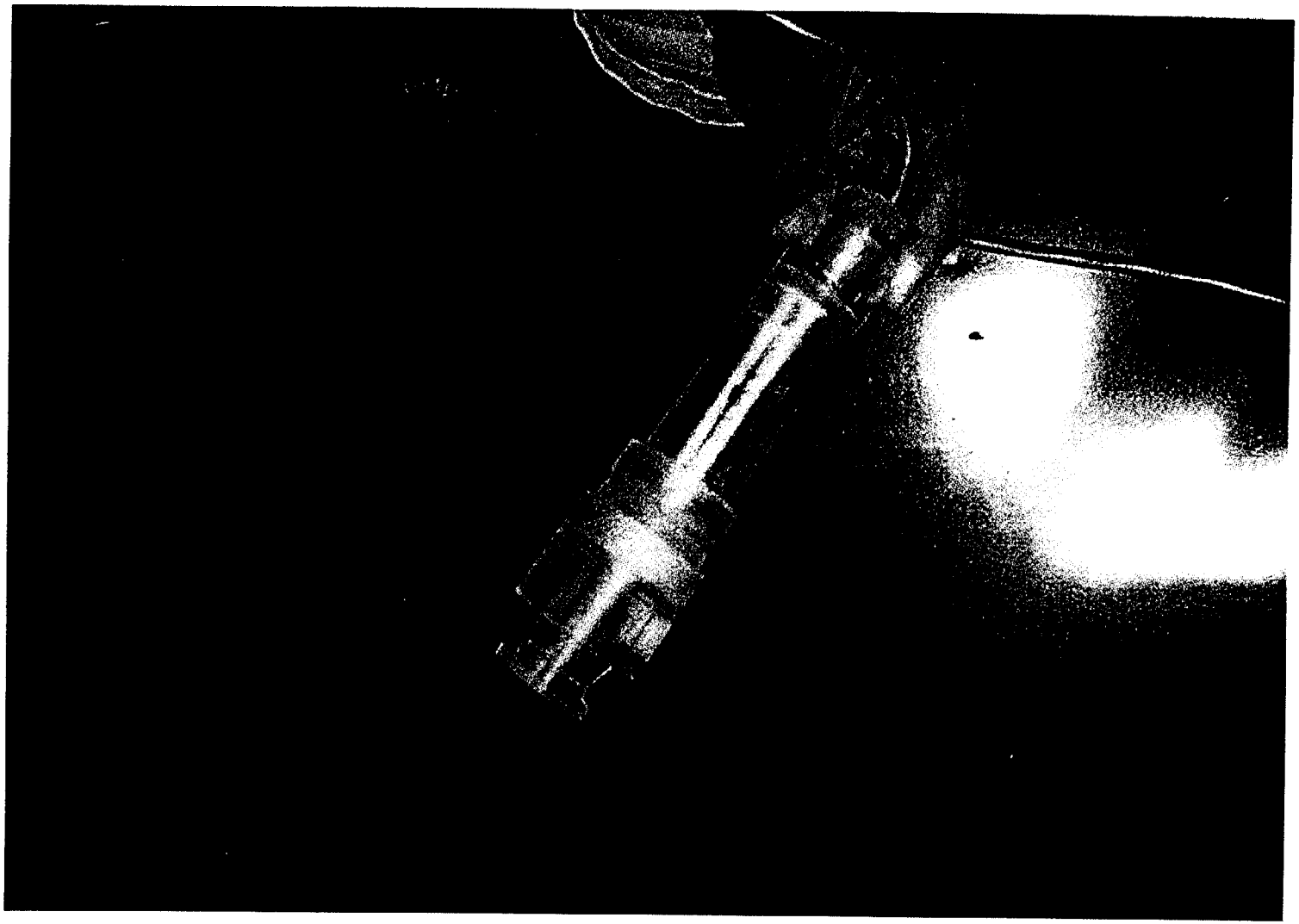
SUBMERSIBLE STRIPPING ANALYZERS

FULLY SUBMERSE AND REMOTELY DEPLOY μTOTAL STRIPPING ANALYZER (performing all sample manipulations in-situ).

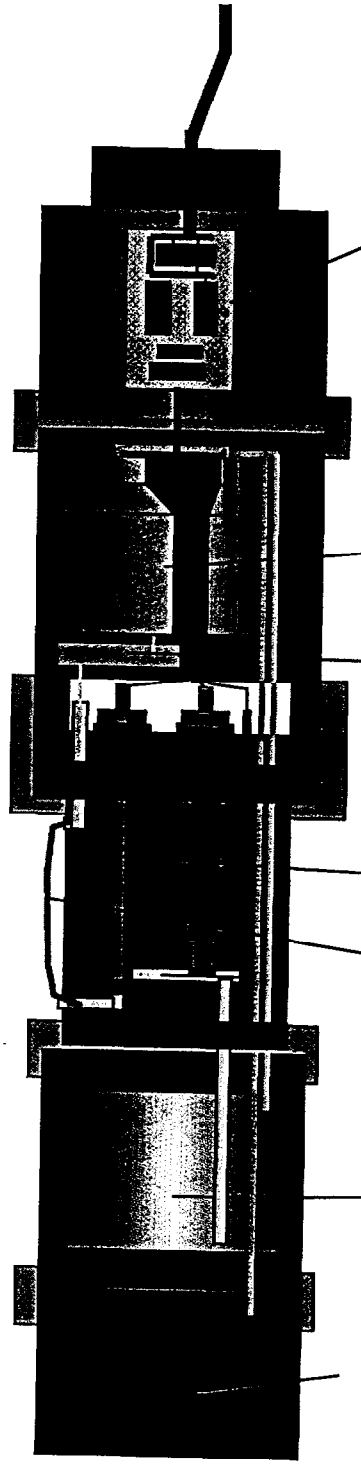
COUPLING THIS in-situ METAL DETECTION CAPABILITY WITH CONTINUOUS MONITORING OF OTHER CHEMICAL AND PHYSICAL PARAMETERS

NMSU REMOTE SENSOR

INTEGERATING THE SENSING ELECTRODES, MICROPUMPING, DIALYSIS SAMPLING, AND BUILD IN ELECTRONIC CONTROL



Integrated Remote Probe



CONCLUSIONS

THE NEW SENSOR TECHNOLOGY WILL REVOLUTIONIZE THE WAY OF MONITORING METAL POLLUTANTS, WILL BRING MAJOR CHANGES TO POLLUTION CONTROL IN DOE FACILITIES, AND WILL SUPPORT THE GOVERNMENT COMMITMENT TO REDUCE THE HUGE COSTS INVOLVED IN THE CHARACTERIZATION OF CONTAMINATED SITES.

(SEE POSTER FOR MORE DETAILS)