

DTIC FILE COPY

4

AD

TECHNICAL REPORT ARCCB-TR-89026

**DETERMINATION OF IRON IN CHROMIUM  
PLATING AND POLISHING SOLUTIONS  
BY ATOMIC ABSORPTION SPECTROMETRY**

AD-A214 467

**SAMUEL SOPOK**

**DTIC**  
**ELECTE**  
**NOV 22 1989**  
**S B D**

**OCTOBER 1989**



**US ARMY ARMAMENT RESEARCH,  
DEVELOPMENT AND ENGINEERING CENTER  
CLOSE COMBAT ARMAMENTS CENTER  
BENÉT LABORATORIES  
WATERVLIET, N.Y. 12189-4050**



**APPROVED FOR PUBLIC RELEASE; DISTRIBUTION UNLIMITED**

89 11 20 055

#### DISCLAIMER

The findings in this report are not to be construed as an official Department of the Army position unless so designated by other authorized documents.

The use of trade name(s) and/or manufacturer(s) does not constitute an official indorsement or approval.

#### DESTRUCTION NOTICE

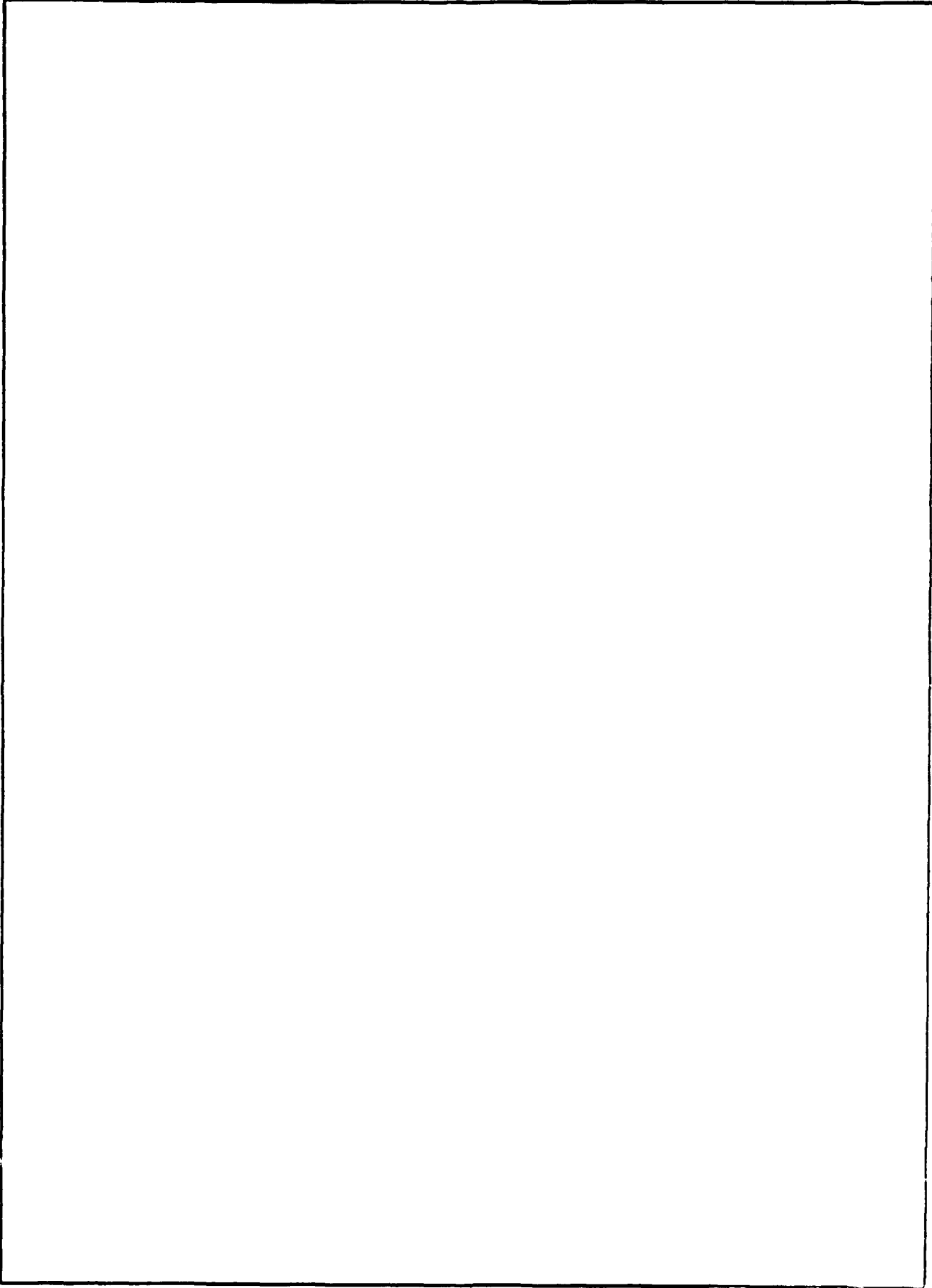
For classified documents, follow the procedures in DoD 5200.22-M, Industrial Security Manual, Section II-19 or DoD 5200.1-R, Information Security Program Regulation, Chapter IX.

For unclassified, limited documents, destroy by any method that will prevent disclosure of contents or reconstruction of the document.

For unclassified, unlimited documents, destroy when the report is no longer needed. Do not return it to the originator.

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER ARCCB-TR-89026	2. GOVT ACCESSION NO.	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) DETERMINATION OF IRON IN CHROMIUM PLATING AND POLISHING SOLUTIONS BY ATOMIC ABSORPTION SPECTROMETRY		5. TYPE OF REPORT & PERIOD COVERED Final
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) Samuel Sopok		8. CONTRACT OR GRANT NUMBER(s)
9. PERFORMING ORGANIZATION NAME AND ADDRESS U.S. Army ARDEC Benet Laboratories, SMCAR-CCB-TL Watervliet, NY 12189-4050		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS AMCMS No. 6126.23.1BLO.0 PRON No. 1A92ZNACNMSC
11. CONTROLLING OFFICE NAME AND ADDRESS U.S. Army ARDEC Close Combat Armaments Center Picatinny Arsenal, NJ 07806-5000		12. REPORT DATE October 1989
		13. NUMBER OF PAGES 10
14. MONITORING AGENCY NAME & ADDRESS (if different from Controlling Office)		15. SECURITY CLASS. (of this report)  UNCLASSIFIED
		15a. DECLASSIFICATION/DOWNGRADING SCHEDULE
16. DISTRIBUTION STATEMENT (of this Report)  Approved for public release; distribution unlimited.		
17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)		
18. SUPPLEMENTARY NOTES  Submitted to <u>Plating and Surface Finishing</u> .		
19. KEY WORDS (Continue on reverse side if necessary and identify by block number) Chemical Analysis Iron Chromium Plating Solutions Polishing Solutions Atomic Absorption Spectrometry.		
20. ABSTRACT (Continue on reverse side if necessary and identify by block number)  The chemical literature lacks a specific analytical method for adequately monitoring iron in chromium plating and polishing solutions during the plating and polishing processes. In this report, a specific method is presented for analyzing and monitoring iron during these processes. The optimum operating range of the iron is generally around 10 g/l maximum in both the chromium plating and the polishing solutions. The resulting precisions are in the range of 0.5 to 1.5 g/l, providing adequate monitoring of these solutions supported by six years of testing.		

SECURITY CLASSIFICATION OF THIS PAGE(When Data Entered)



SECURITY CLASSIFICATION OF THIS PAGE(When Data Entered)

TABLE OF CONTENTS

	<u>Page</u>
ACKNOWLEDGEMENTS .....	ii
INTRODUCTION .....	1
EXPERIMENTAL PROCEDURE .....	1
RESULTS AND DISCUSSION .....	2
REFERENCES .....	4

TABLES

I. STANDARD SOLUTION DATA FOR IRON .....	5
II. EXPERIMENTAL SAMPLE SOLUTION DATA FOR IRON IN CHROMIUM PLATING SOLUTIONS .....	5
III. EXPERIMENTAL SAMPLE SOLUTION DATA FOR IRON IN POLISHING SOLUTIONS .....	5
IV. PRECISION OF MICROPIPETTING 0.500 ml .....	6
V. PRECISION OF MICROPIPETTING 0.250 ml .....	6
VI. PRECISION OF MICROPIPETTING 0.050 ml .....	7
VII. PRECISION OF A 100-ml CLASS-A VOLUMETRIC FLASK .....	7
VIII. PRECISION OF A 1-g/l IRON STANDARD SOLUTION .....	8
IX. PRECISION OF A 5-ppm IRON STANDARD SOLUTION BY AA SPECTROMETRY .....	8

<b>Accession For</b>	
NTIS GRA&I	<input checked="" type="checkbox"/>
DTIC TAB	<input type="checkbox"/>
Unannounced	<input type="checkbox"/>
Justification	
By _____	
Distribution/	
Availability Codes	
	Avail and/or Special
A-1	

## ACKNOWLEDGMENTS

Special thanks are given to Ellen Fogarty and Rose Neifeld of Benet Laboratories for their respective word processing and technical editing work on this manuscript.

## INTRODUCTION

Iron is produced as an unwanted by-product of polishing and chromium plating solutions for low alloy steels (refs 1-3). The chemical literature lacks a specific analytical method for adequately monitoring iron in chromium plating and polishing solutions during the plating and polishing processes. Lack of optimization of these plating and polishing solutions causes serious problems for the chromium plating industry such as poor quality products, wasted human resources, and wasted electrical energy.

A common chemical analysis method to determine iron in the presence of chromium plating and polishing solutions is composed of an alkaline precipitation of iron (ref 4). This method provides adequate precisions, but an unacceptable analysis time of two days.

The specific method given in this report provides both acceptable analysis and monitoring of iron in chromium plating and polishing solutions. This method consists of atomic absorption (AA) spectrometry (ref 5).

## EXPERIMENTAL PROCEDURE

Strict analytical chemistry methods and procedures are followed throughout this experimental procedure section. An excellent source of reference for these methods and procedures is by Fritz and Schenk (ref 4).

One analytical reagent grade standard solution is required. It is a  $1.000 \pm 0.005$ -g/l iron solution also containing 50 milliliters (ml) of concentrated nitric acid per liter that meets American Chemical Society (ACS) and American Society For Testing and Materials (ASTM) Standards (ref 6).

---

References are listed at the end of this report.

Tables II and III present the sample solution data for iron in chromium plating and polishing solutions, respectively. These sample solutions are diluted 1:2000 in order to attain detector linearity. Due to a linear operating range, the following simplified calculation is used to determine iron concentration in the original chromium plating and polishing sample solutions:

$$\text{g/l Iron} = (10)(\text{sample absorbance}/5 \text{ ppm standard absorbance})$$

The chromium plating sample solution in Table II has a 2.72-g/l iron concentration, and the polishing sample solution in Table III has a 5.44-g/l iron concentration.

Chromium plating solutions typically contain 240 to 260 g/l chromic acid and 2.4 to 3.0 g/l sulfuric acid, while polishing solutions typically contain 640 to 730 g/l phosphoric acid and 795 to 895 g/l sulfuric acid. The major components of these sample solutions do not interfere with the iron determination by AA spectrometry (ref 6).

It is useful to evaluate the variations in precision for the materials and methods used. Tables IV through VIII present these data for the 0.500-ml micropipets, 0.250-ml micropipets, 0.050-ml micropipets, the 100-ml class-A volumetric flasks, and the 1-g/l iron standard solution, respectively. Variations in precision are also evaluated for the AA spectrometer. Table IX presents these data for six consecutive replicates of the 5-ppm iron standard solution.

The data obtained by this specific method are sufficient to adequately monitor the iron in chromium plating and polishing processes, thus providing efficient use of resources. The optimum operating range of iron is generally around 10 g/l maximum, and the resulting precisions are in the range of 0.5 to 1.5 g/l, providing adequate monitoring of these solutions supported by six years of testing.



## REFERENCES

1. K. Langford and J. Parker, Analysis of Electroplating and Related Solutions, Metals and Plastics Publications, Inc., Hackensack, NJ, 1986.
2. T. Irvine, The Chemical Analysis of Electroplating Solutions: A Theoretical Approach, Chemical Publishing Company, New York, 1970.
3. Metal Finishing Guidebook, Metals and Plastics Publications, Inc., Hackensack, NJ, 1984.
4. J. Fritz and G. Schenk, Quantitative Analytical Chemistry, Fifth Edition, Allyn and Bacon, Inc., Boston, MA, 1987.
5. H. Bauer, G. Christian, and J. O'Reilly, Instrumental Analysis, Allyn and Bacon, Inc., Boston, MA, 1978.
6. "AA/ICP Operators Manual," Perkin-Elmer Corp., Norwalk, CT, 1981.

TABLE I. STANDARD SOLUTION DATA FOR IRON

Replicate	Absor. (AU) 0.00 ppm Iron	Absor. (AU) 2.50 ppm Iron	Absor. (AU) 5.00 ppm Iron
1	0.003	0.100	0.197
2	0.002	0.104	0.200
3	0.002	0.098	0.202
X(avg)	0.002	0.101	0.200

TABLE II. EXPERIMENTAL SAMPLE SOLUTION DATA FOR IRON IN CHROMIUM PLATING SOLUTIONS

Replicate	Sample Iron Absor. (AU)	Sample Iron Conc. (ppm)
1	0.052	1.30
2	0.055	1.38
3	0.056	1.40
X(avg)	0.054	1.36

TABLE III. EXPERIMENTAL SAMPLE SOLUTION DATA FOR IRON IN POLISHING SOLUTIONS

Replicate	Sample Iron Absor. (AU)	Sample Iron Conc. (ppm)
1	0.112	2.80
2	0.108	2.70
3	0.106	2.65
X(avg)	0.109	2.72

TABLE IV. PRECISION OF MICROPIPETTING 0.500 ml

Replicate	Volume (ml)*
1	0.5026
2	0.5115
3	0.5118
4	0.5013
5	0.5054
6	0.5079
X(avg)	0.5068
Sn	0.0044

\*Volumes are calculated from the weight-volume relationship of each micropipetted deionized water solution corrected for temperature.

TABLE V. PRECISION OF MICROPIPETTING 0.250 ml

Replicate	Volume (ml)*
1	0.2582
2	0.2497
3	0.2546
4	0.2545
5	0.2557
6	0.2532
X(avg)	0.2543
Sn	0.0028

\*Volumes are calculated from the weight-volume relationship of each micropipetted deionized water solution corrected for temperature.

TABLE VI. PRECISION OF MICROPIPETTING 0.050 ml

Replicate	Volume (ml)*
1	0.0494
2	0.0508
3	0.0533
4	0.0522
5	0.0497
6	0.0517
X(avg)	0.0512
Sn	0.0015

\*Volumes are calculated from the weight-volume relationship of each micropipetted deionized water solution corrected for temperature.

TABLE VII. PRECISION OF A 100-ml CLASS-A VOLUMETRIC FLASK

Replicate	Volume (ml)*
1	100.14
2	99.97
3	99.89
4	100.12
5	100.03
6	100.06
X(avg)	100.04
Sn	0.09

\*Volumes are calculated from the weight-volume relationship of the contained deionized water solution corrected for temperature.

TABLE VIII. PRECISION OF A 1-g/l IRON STANDARD SOLUTION

Replicate	Iron Conc. (g/l)*
1	1.003
2	1.000
3	1.002
4	1.001
5	1.002
6	1.002
X(avg)	1.002
Sn	0.001

\*Iron concentrations are determined by the alkaline precipitation method in Fritz and Schenk (ref 4) which is a standard chemical analysis method for iron.

TABLE IX. PRECISION OF A 5-ppm IRON STANDARD SOLUTION BY AA SPECTROMETRY

Replicate	Absor. (AU) 5.00 ppm Iron
1	0.201
2	0.198
3	0.203
4	0.200
5	0.200
6	0.204
X(avg)	0.201
Sn	0.002

TECHNICAL REPORT INTERNAL DISTRIBUTION LIST

	<u>NO. OF COPIES</u>
CHIEF, DEVELOPMENT ENGINEERING DIVISION	
ATTN: SMCAR-CCB-D	1
-DA	1
-DC	1
-OM	1
-DP	1
-DR	1
-DS (SYSTEMS)	1
CHIEF, ENGINEERING SUPPORT DIVISION	
ATTN: SMCAR-CCB-S	1
-SE	1
CHIEF, RESEARCH DIVISION	
ATTN: SMCAR-CCB-R	2
-RA	1
-RM	1
-RP	1
-RT	1
TECHNICAL LIBRARY	5
ATTN: SMCAR-CCB-TL	
TECHNICAL PUBLICATIONS & EDITING SECTION	3
ATTN: SMCAR-CCB-TL	
DIRECTOR, OPERATIONS DIRECTORATE	1
ATTN: SMCWV-OD	
DIRECTOR, PROCUREMENT DIRECTORATE	1
ATTN: SMCWV-PP	
DIRECTOR, PRODUCT ASSURANCE DIRECTORATE	1
ATTN: SMCWV-QA	

NOTE: PLEASE NOTIFY DIRECTOR, BENET LABORATORIES, ATTN: SMCAR-CCB-TL, OF ANY ADDRESS CHANGES.

TECHNICAL REPORT EXTERNAL DISTRIBUTION LIST

	<u>NO. OF COPIES</u>		<u>NO. OF COPIES</u>
ASST SEC OF THE ARMY RESEARCH AND DEVELOPMENT ATTN: DEPT FOR SCI AND TECH THE PENTAGON WASHINGTON, D.C. 20310-0103	1	COMMANDER ROCK ISLAND ARSENAL ATTN: SMCRI-ENM ROCK ISLAND, IL 61299-5000	1
ADMINISTRATOR DEFENSE TECHNICAL INFO CENTER ATTN: DTIC-FDAC CAMERON STATION ALEXANDRIA, VA 22304-6145	12	DIRECTOR US ARMY INDUSTRIAL BASE ENGR ACTV ATTN: AMXIB-P ROCK ISLAND, IL 61299-7260	1
COMMANDER US ARMY ARDEC ATTN: SMCAR-AEE	1	COMMANDER US ARMY TANK-AUTMV R&D COMMAND ATTN: AMSTA-DDL (TECH LIB) WARREN, MI 48397-5000	1
SMCAR-AES, BLDG. 321	1	COMMANDER	
SMCAR-AET-O, BLDG. 351N	1	US MILITARY ACADEMY	1
SMCAR-CC	1	ATTN: DEPARTMENT OF MECHANICS	
SMCAR-CCP-A	1	WEST POINT, NY 10996-1792	
SMCAR-FSA	1		
SMCAR-FSM-E	1	US ARMY MISSILE COMMAND	
SMCAR-FSS-D, BLDG. 94	1	REDSTONE SCIENTIFIC INFO CTR	2
SMCAR-IMI-I (STINFO) BLDG. 59	2	ATTN: DOCUMENTS SECT, BLDG. 4484	
PICATINNY ARSENAL, NJ 07806-5000		REDSTONE ARSENAL, AL 35898-5241	
DIRECTOR US ARMY BALLISTIC RESEARCH LABORATORY ATTN: SLCBR-DD-T, BLDG. 305	1	COMMANDER US ARMY FGN SCIENCE AND TECH CTR ATTN: DRXST-SD 220 7TH STREET, N.E. CHARLOTTEVILLE, VA 22901	1
DIRECTOR US ARMY MATERIEL SYSTEMS ANALYSIS ACTV ATTN: AMXSY-MP	1	COMMANDER US ARMY LABCOM MATERIALS TECHNOLOGY LAB ATTN: SLCMT-IML (TECH LIB)	2
ABERDEEN PROVING GROUND, MD 21005-5071		WATERTOWN, MA 02172-0001	
COMMANDER HQ, AMCCOM ATTN: AMSMC-IMP-L	1		
ROCK ISLAND, IL 61299-6000			

NOTE: PLEASE NOTIFY COMMANDER, ARMAMENT RESEARCH, DEVELOPMENT, AND ENGINEERING CENTER, US ARMY AMCCOM, ATTN: BENET LABORATORIES, SMCAR-CCB-TL, WATERVLIET, NY 12189-4050, OF ANY ADDRESS CHANGES.

TECHNICAL REPORT EXTERNAL DISTRIBUTION LIST (CONT'D)

	<u>NO. OF COPIES</u>		<u>NO. OF COPIES</u>
COMMANDER US ARMY LABCOM, ISA ATTN: SLCIS-IM-TL 2800 POWDER MILL ROAD ADELPHI, MD 20783-1145	1	COMMANDER AIR FORCE ARMAMENT LABORATORY ATTN: AFATL/MN EGLIN AFB, FL 32542-5434	1
COMMANDER US ARMY RESEARCH OFFICE ATTN: CHIEF, IPO P.O. BOX 12211 RESEARCH TRIANGLE PARK, NC 27709-2211	1	COMMANDER AIR FORCE ARMAMENT LABORATORY ATTN: AFATL/MNF EGLIN AFB, FL 32542-5434	1
DIRECTOR US NAVAL RESEARCH LAB ATTN: MATERIALS SCI & TECH DIVISION CODE 26-27 (DOC LIB) WASHINGTON, D.C. 20375	1 1	METALS AND CERAMICS INFO CTR BATTELLE COLUMBUS DIVISION 505 KING AVENUE COLUMBUS, OH 43201-2693	1

NOTE: PLEASE NOTIFY COMMANDER, ARMAMENT RESEARCH, DEVELOPMENT, AND ENGINEERING CENTER, US ARMY AMCCOM, ATTN: BENET LABORATORIES, SMCAR-UCCB-TL, WATERVLIET, NY 12189-4050, OF ANY ADDRESS CHANGES.