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DNA-TR-92-182

Commercial Products from Demilitarization Operations

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May 1993

Technical Report

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13. ABSTRACT (Maximum 200 words) This report surveys technical literature for processes which could be used to convert Russian chemical agents to useful products. Possible reactions to explain a Russian process for the conversion of Lewisite to Arsenic are presented. A short reference on the economic viability of high-purity Arsenic production is provided. A Westinghouse process is offered as a possible solution to the Russian problem.			
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CONVERSION TABLE

Multiply ----- by ----- > To Get		
To Get <----- by ----- Divide		
Kilogram (kg)	2.2	pounds (lb.)
tonne (1,000 kg)	1.1	short ton
°C (Celsius temperature)	$9/5 (°C) + 32$	°F (Fahrenheit temperature)

Accession For	
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Unannounced	<input type="checkbox"/>
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SECTION 1

TASKING

Conduct research to examine possible commercial products which could be made from the demilitarization of CW agents.

SECTION 2

BACKGROUND

Russia has requested U.S. assistance in the complete destruction of all chemical warfare munitions and bulk agents. The Russian Federation announced that it was exploring for feasible technologies for the conversion of CW agents into useful commercial products.^{3,2} This effort was made to comply with the provisions of the CWC but may also have been intended as a sop to the Russian people both to gain popular support for what was perceived to be an environmentally unpopular activity and to provide some economic offset for the destruction process. In a meeting in June 1992, between U.S. and Russian officials, the Russians outlined several proposals for the destruction of CW materials.⁴

SECTION 3

COMMERCIAL PRODUCTS

While there is always the possibility that the Russians may chose to incinerate or otherwise destroy all stockpiled chemical agents, their representatives have indicated an interest in exploring processes for the conversion of these agents to marketable products and have proposed an as yet unspecified way of extracting high-grade arsenic from Lewisite.

The Russians envisioned the use of arsenic in the production of Gallium Arsenide for high speed computer chips because of the purported price of high-grade arsenic (\$4500/kg).¹ A Battelle white paper (Appendix B) analyzed the status of the market for high-grade arsenic and the marketability of this process and concluded that the current market value for high-grade arsenic is more than an order of magnitude less than the Russian evaluation. Further, the advantage of increased speed of the Gallium Arsenide chip in serial processors can be offset by the use of much cheaper silicon-based chips in parallel processors, undermining economic arguments for this process.

Other uses for arsenic are as a wood preservative (in the U.S. this comprises about 65% of the demand for arsenic), in pesticides (20% of the U.S. arsenic demand) and for specialized glasses.² Arsenic can be converted to chromated copper arsenate for use as a wood preservative in pressure-treated lumber. This usage could take advantage of the vast Russian forest preserve and does not require high-purity arsenic. Use of arsenic in pesticides, specifically for the cotton industry, is scheduled to be phased out in the U.S.¹⁰ An annotated bibliography of papers on the conversion of CW agents to marketable products is provided at Appendix A.

SECTION 4

LEWISITE CONVERSION

The Russian-American University reported sponsorship of an experimental process for the production of arsenic from Lewisite.⁴ The Russians proposed to convert Lewisite to high-purity arsenic through a process which involves hydrolysis with aqueous sodium hydroxide to produce acetylene and an aqueous phase containing sodium salts of arsenic. Electrolysis of the aqueous phase would then be used to generate arsenic metal. The Russians were unwilling to provide any details on the electrolysis process until they had it patented. We have, however, learned that their electrolysis process involves a sodium metaarsenate-sodium chloride solution and yields chlorine and oxygen gases at one electrode, arsenic and hydrogen gas at the other and sodium hydroxide in the cell. The process purports to yield 99.99% pure arsenic (insufficient for chip grade material), but since the Russians are reported to be using AsCl_3 reagent as their starting material for the test cell and not Lewisite itself, this figure may be optimistic.

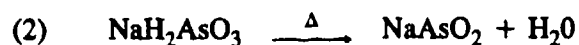
In an attempt to explain how the Russian process might work, we have examined chemical reactions by which Lewisite could be converted to arsenic. Some of this chemistry involves well established industrial processes and reactions; others have only been reported in research literature; a few are theoretically possible based on analogous reactions for elements in the same chemical family (i.e., phosphorus).

The following equations describe a possible path for the Lewisite to arsenic process:

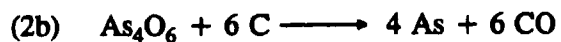


Lewisite

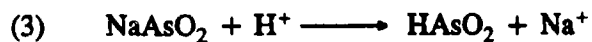
Step (1) describes the hydrolysis of Lewisite with sodium hydroxide under controlled pH. Removed acetylene gas may be burned off. This is likely the hydrolysis process described by the Russians and involves known chemistry.⁴



In Step (2), Sodium metaarsenite can be formed by heating the arsenate and driving off water. This reaction has been reported in the chemical literature for arsenic and Phosphorus compounds.^{9,7}



In step (2a), the metaarsenite from step (2) can dissociate to produce As_4O_6 , a dimer of arsenic trioxide.¹³ Roasting the trioxide over charcoal (step (2b)) involves established technology for reducing the oxide to arsenic metal.⁷



Step (3) is the conversion of sodium metaarsenite to metaarsenious acid.



Step (4) shows the electrochemical reduction of metaarsenious acid in sulfuric acid at pH 2.¹²

SECTION 5

PREPARATION OF HIGH-PURITY ARSENIC

A number of methods have been devised for the preparation of arsenic of sufficient purity to qualify for chip applications. Among these are a process involving distillation of a meld of high-purity bismuth and commercial (about 99% pure) arsenic under vacuum to yield arsenic of greater than 99.999% purity.¹⁵ 99.99% pure arsenic has been produced directly from the trioxide dimer by reaction with HCl and H₂SO₄ at about 600°C.¹⁴ Catalytic methods have also been developed for the preparation of 99.99% arsenic.⁶

Westinghouse has developed a proprietary process which involves the use of a plasma torch to pyrolyze materials at 5000 to 7000°C, converting them into their elemental forms or allowing them to react under controlled chemical conditions to other species. This process is currently being used by industry to recover scrap metals and has been tested in the pyrolysis of PCBs with an extremely high conversion rate (reported up to 8-9's). Westinghouse indicated that the Russians have indicated interest in exploring using the plasma torch to convert Lewisite to AsCl₃ or further, into metallic arsenic. Thus far Westinghouse has only conducted theoretical thermodynamic studies on the Lewisite conversion process.¹¹

SECTION 6

CONCLUSION

None of the chemical processes described in this paper have yet been commercially exploited. While each of the individual steps described above is feasible, the engineering and scale up of the process to a production level may be prohibitively difficult. More importantly, unless other metallic impurities which are likely to be found in Lewisite are removed, the high purity required for chip application may require additional steps. If potential corrosion and other design problems could be overcome, the Westinghouse process may provide the most viable solution to the Russian Federation recovery problem.

SECTION 7

LIST OF REFERENCES

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4. **Meeting Notes, OTASD(AE)(CM) 3 June, Moscow, Subject: Russian Federation Technical Concepts for CW Destruction. (Unclassified)**
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14. **Toyabe. et. al., High Purity Arsenic Metal Production, (Unclassified) Arsenic Metallurgy Fundamentals and Applications, 1987. (Unclassified)**

15. Yokozawa, M., Preparation of High-purity Arsenic, (Unclassified) U.S. Pat 3512758, 19 May 1970. (Unclassified)

Appendix A

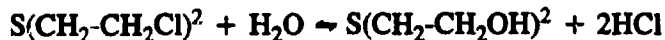
Annotated Bibliography: Processes for the Conversion of Chemical Warfare Agents to Useful Materials.

1. Comments on the Proposed Conversion of Stockpiled CW Agents into Useful Commercial Products, Personal Communication, Dr. Herbert S. Aaron, CRDEC, April 24 1992.

A synopsis of Dr. Aaron's observations based on a document search by the CRDEC Technical Library. The only relevant reference cited was a Thiokol report (Ref. 3) although other reports are cited (Refs 4-5). Dr. Aaron knows of no formal in-house CRDEC projects on this topic (he speculated that any record of CRDEC work on this is buried in unpublished lab notes). Based exclusively on the chemistry involved, Aaron suggests: Mustard can be converted to polysulfide rubbers; Lewisite can be converted into a useful arsenic derivative; the hydrolysis product of G agents can be used in the preparation of polymers and plasticizers; the hydrolysis product of VX can be used in preparing NPK fertilizer; and salt residues can be converted into sulfur or calcium sulfate for other than land disposal.

2. Effects of Water on Sulfur Ethyl Dichloride, Note of Mssrs. Ch. Boulin and L.J. Simon, Minutes of Meeting, French Academy of Sciences, April 6, 1920.

Describes basic research of the conditions under which the hydrolysis of Mustard to thiodiglycol occurs by the reversible equilibrium:



where neutralization of the acid can drive the reaction to the right. No commentary regarding the commercial use of thiodiglycol.

3. Study of the Conversion of Mustard to Useful Products, C. Vriesen, G. Rowell, J. Robbins et.al., Thiokol Corp, Edgewood Arsenal Contractor Report, EM-CR-76062, March 1976.

A thorough literature study of the conversion of Mustard to useful materials including polysulfide polymers and the possible uses of those polymers. Four products with theoretical market potential were identified: 1. mustard polysulfide made from the polymerization of mustards (uses: hoses, coatings, sulfur cements, pipejoining sealers, gaskets, fabric impregnation, sealing aircraft wing tanks, liners for concrete fuel storage tanks, adhesive backings, leather impregnation, valve lubricants and rubber patterns for plaster molds); 2. thiodiglycol from the hydrolysis of mustard (uses: printing ink stabilizer, dye solvent, epoxide cure accelerator, paper strength enhancement, acid resistance, road surface cover, resistance to aliphatic hydrocarbons); 3. divinyl sulfone from the oxidation of mustard through an intermediate sulfone (uses: dye fixative, insecticides, fabric preparation - waterproofing, creaseproofing, sizing, plasticizers, and films); and 4. bis (2-hydroxyethyl) sulfone also from oxidation or hydrolysis (uses: dye fastness, textile

finishing). Other possible products involving replacement of the terminal chlorine with various substituents may have some application in nickel plating or heavy metal scavenging, but have not been commercially explored. The mustard polysulfide was determined to be the most useful application based on ease of conversion, the relative difficulty of reconversion, the completeness of the conversion reaction (little Mustard left), best market development, and least vesicant property.

4. Treatment and Disposal of Future Demil Residues, Bruce Carr, report AAI-ER-8956, April 1977. Distribution restricted.

5. Decontamination and Disposal of Chemical Agents (original in German, translation under FSTC-HT-947-84, January 1985), Karlheinz Lohs and Dieter Martinetz, GDR Patent 146 P 1983.

Although principally a treatise on demilitarization, Lohs' paper devotes a chapter to identifying possible commercial uses of CW agent products. He identifies possible uses for mustard derivatives in the fabric industry and in fungicides (from divinyl sulphone) and for the conversion of GA, GB to pesticides. Few details.

6. Demilitarization of Mustard Agent by Conversion to a Polysulfide Polymer, S. Kessler, C.W. Vriesen, Thiokol Corp., Edgewood Arsenal Contractor Report EM-CR-77012, October 1976.

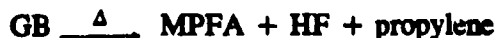
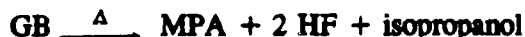
Describes work for Pine Bluff Arsenal to set up bench scale conversion of HD Mustard to polysulfide rubber and to convert the crude latex to useable forms. Although high conversion efficiency to the polysulfide was cited (as little as .05 ppm of Mustard remained), polymer utility was limited to flexible putty compositions as sealers in buildings, in boat and aircraft construction and in aircraft wing fuel tanks. The polymer might also be used in linings for paint, oil and gasoline hoses. The polysulfide could not be converted to a liquid polysulfide (a process reported in other references) and would not mix with asphalt to make road surface compositions. It was suggested that mixing polysulfides can be used in the preparation of sulfur cements as well as pipejoining sealing compounds by analogy with several patented processes.

7. GB-DF Conversion Studies, Vol. 1, Chemical Approaches to Conversion, T. M. Prociw, R.E. Wyant, T.B. Stanford, H.E. Thompson, R.K. Smith, Battelle, U.S. Army Toxic and Hazardous Materials Agency Report DRXTH-TE-CR-81109, February 1981.

Five out of fifteen possible approaches for the conversion of GB to DF were selected. While no commercial applications are cited, this reference identifies intermediates which may have commercial use. For example, in the acid-catalyzed hydrolysis of GB, methylphosphonic acid is an intermediate. A 1980 SIPRI review recommends the conversion of GB to methylphosphonic acid as a precursor to various pesticides (see ref 9).

8. GB-DF Conversion Studies, Vol. 2, Process Engineering Recommendations, L.M. Curran, T. M. Prociw, R.K. Smith, T.B. Stanford, J.A. Telljohann, Battelle, U.S. Army Toxic and Hazardous Materials Agency Report DRXTH-TE-CR-81110, June 1981.

This report is an engineering elaboration of the two most viable processes cited in ref 7 above which produce either isopropanol or propylene according to the following:



(where MPA = methylphosphonic acid, MPFA = methylphosphono-fluoridic acid, and the delta symbol indicates a heating process). The report identifies equipment required, approximate per pound costs (\$.09 and \$.05 respectively for the above), theoretical yields (4070 lb. of isopropanol or 2860 lb. of propylene from 9500 lb. of GB) and waste disposal for the processes.

9. Destruction or Conversion of Chemical Warfare Agents: Possibilities and Alternatives, Lohs, K.; Chemical Weapons: Destruction and Conversion pps. 67-75, Stockholm International Peace Research Institute, Crane & Russak Co.Inc., N.Y. 1980.

Presents arguments for the conversion of CW agents and intermediates to materials with application in civilian industries such as pesticides, pharmaceuticals, dyestuffs, and plastics as an alternative to their destruction. Identifies reactions for the conversion to intermediate materials, but does not suggest processes for the conversion of these intermediates to final products.

10. Analysis of Foreign Organophosphorus Chemical Technology, T.L. Ferguson, A.R. Hylton, C.E. Mumma, ACDA ST-197, Volume III, Midwest Research Institute, 13 Nov. 1972.

Describes a process for the preparation of a class of pesticides from acid chlorides and alcohols in the presence of a Lewis acid acceptor. Thus, dichlor could be converted to through an intermediate $\text{Cl}(\text{RO})\text{P}(\text{O})\text{Me}$, into a pesticide, $(\text{RO})\text{P}(\text{O})(\text{SR}')\text{Me}$ (where R and R' are alkyl groups). British Patent 797,603 was issued regarding this process.

Appendix B

ARSENIC

COMMODITY

Arsenic is most commonly used in one of three forms: arsenic trioxide (As_2O_3), arsenic metal, and high purity arsenic, (greater than 99.99% purity). In the United States, 70% of the demand for arsenic is for chromated copper arsenate which is used as a wood preservative in pressure treated lumber. About 20% of the demand is for agricultural chemicals, principally for the desiccation of cotton. The production of pressed and blown glass accounts for about 4% of the demand and 2% for non-ferrous alloys. The remaining demand is for high purity arsenic for use in the production of optical glass and in electronics applications such as gallium arsenide semi-conductors. The Russians also cite the use of arsenic as a lead additive in gunshot casting, a component of type casting for printing, and for the manufacture of chalcocite (Cuprous Sulfide) and optical heat resistance glass.

PRODUCTION

The production of arsenic trioxide is generally the byproduct of the smelting and refining of lead, copper, and gold. The ores are roasted and arsenic trioxide is recovered by the sublimation onto cooler surfaces or by electrostatic precipitation. The recovery of arsenic is driven as much by environmental concerns as for its commodity value. The production of metallic arsenic is by the reduction of As_2O_3 . Metallic arsenic represents only 3% of the world wide demand for arsenic. High purity arsenic is produced by reduction roasting with charcoal.

SOURCES

The number of world-wide sources for arsenic and its compounds has decreased due to environmental regulation and associated costs and the actions in the market of China. The production of arsenic and its compounds has ceased entirely in the United States and in Sweden due to environmental concerns. Production in other countries has been curtailed due to the dominance of the market by China in the last two years. China, and to a lesser extent Chile, have driven other producers out of the As_2O_3 and metal market (99.99% purity metal) by keeping their prices very low. Note that the Chinese sell only arsenic trioxide and metallurgical grade metal and none of the high purity metal. The three principle sources of high purity arsenic metal, greater than 99.999% purity, are Japan, 30 metric tonne capacity, Germany, 15 metric tonne capacity, and Canada.

MARKET

The world-wide market for arsenic and its compounds has been forecast by the United States Bureau of Mines as declining. The trend for arsenic trioxide has been a 24.5% reduction in world production over the last five years. The demand is now less than 50 thousand metric tonnes per year for arsenic trioxide. The estimate for high purity arsenic metal is less than 50 metric tonnes

per year. There is no United States Government program for the stockpiling of arsenic. Estimates for gallium arsenide demand have been downsized from earlier estimates due to the advent of parallel processing in computer architectures. The increased computing speed of the gallium arsenide computer chip has been offset by the cheaper silicon based chip used in parallel. The growth in gallium arsenide production capacity world-wide has also mitigated against a significant pressure on the supply.

PRICES

Recent market prices for arsenic trioxide are around \$00.36/lb or approximately \$00.79/kilogram by the carload lot. Metallic arsenic from China has been fluctuating but is in the range of \$1.00 to \$2.00/lb or approximately \$2.20 to 4.40/kilogram. High purity arsenic for electronic use is \$185-220/kilogram on the western market. Polycrystal gallium arsenide (99.99999% purity) is approximately \$1.00/gram while a finished 4 inch polished gallium arsenide wafer ranges from \$280 to \$375 per wafer. Catalog prices for gallium arsenide have been seen which are significantly higher, however, these prices do not reflect the current commercial market price.

FORECAST

The world-wide demand for all arsenic is expected to continue to decline. This is due primarily to the increasingly stringent requirements for pollution control on the production and use of arsenic bearing products. Higher levels of pollution control on the lead, copper, and gold refining processes may actually increase the recovery of arsenic and increase the available supply. The estimates of the mid-1980's for a much higher demand for high purity arsenic for use in electronics and optics has not occurred partially due to substitution and partially due to new computer architectures. The use of arsenic in agricultural applications has been significantly reduced and substitution of arsenic by organic compounds is expected. The only projected growth areas for the use of arsenic is in the treatment of lumber products and that is dependent upon a growth in housing starts.

ANALYSIS

Translations of published reports in the Russian press describe a plan to extract "high extra pure arsenic" from Lewisite and sell the arsenic on the world market. The claims made in the reports for very high prices on the world market for "extra pure" arsenic are not in line with current market prices reported by the Bureau of Mines and by industry sources. Russian contacts with US commodity brokers have also cited unusually high prices.

Plausible explanations for the discrepancies between the perceived Russian value of high purity arsenic are as follows:

- a. The Russians are using inflated catalog prices, i.e. Johnson Matthey, and then extrapolating inflation and/or rapid market growth outstripping supply.

- b. Translations from the Russian to English may be transposing the word dollars for rubles and not making the numeric conversion. The official conversion rate may exceed 600 rubles to 1 US dollar.
- c. There may be confusion in the translations between high purity arsenic and gallium arsenide, or the Russians may have transposed the market price for some form of finished gallium arsenide to high purity arsenic.

The extraction of arsenic from Lewisite raises some interesting considerations:

- a. The Russian Lewisite is 40 years old and is 30% arsenic by their account.
- b. The residual will be $70\% \times 6700$ metric tonnes = 4690 tonnes + reactants. This residual will have to be disposed of.
- c. At current western market rates for high purity arsenic, i.e. \$200/kg, the most the Russians could expect to realize from the sale would be $30\% \times 6700$ tonnes = 2010 tonnes \times \$200,000/tonne = \$402M \times 120 rubles/dollar = 48.240 B rubles.
- d. The 48 billion rubles would be the gross return on the sale of the high purity arsenic. From the gross sales one must subtract the cost of conversion and the cost of disposal of the remaining volume of the former Lewisite and the reactants.
- e. Industry sources are very skeptical of Russian assays and do not believe that the Russians can deliver true high purity arsenic greater than 99.99% purity.

We note that the Russians have approached US brokers with "high purity" arsenic direct from the manufacturing facility in Tblisi.

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OFFICE OF ASSISTANT SECRETARY OF ARMY RESEARCH, DEVELOPMENT & ACQ

ATTN: COL ADAMS

U S ARMY CHEMICAL SCHOOL

ATTN: ATZN-CM-FTO MR SHEHEANE

U S ARMY MATERIAL COMMAND

ATTN: AMCAN-CN COL NIKAI

USA CML & BIOLOGICAL DEFENSE AGENCY

ATTN: AMSCB-TC MR COLBURN
2 CY ATTN: MR FLAMM
5 CY ATTN: SMCCR-MUM DR HUTCHINSON

DEPARTMENT OF THE NAVY

ASSISTANT SECRETARY OF THE NAVY

ATTN: MR GRATTON

OFFICE OF CHIEF OF NAVAL OPERATIONS

ATTN: OP-65 CAPT JOHNSON

DEPARTMENT OF THE AIR FORCE

USAF/XOXXI

ATTN: COL ENGSTROM

DEPARTMENT OF ENERGY

DEPARTMENT OF ENERGY

ATTN: DP 5.1 MS CASEY
ATTN: DP-5.1 G DUDDER

OTHER GOVERNMENT

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ATTN: DR SEIDERS
ATTN: MR MIKULAK
ATTN: MR STAPLES

CENTRAL INTELLIGENCE AGENCY

ATTN: MR SPALDING

DEPARTMENT OF DEFENSE CONTRACTORS

BATTELLE MEMORIAL INSTITUTE

2 CY ATTN: S BOSCO

KAMAN SCIENCES CORP

ATTN: DASIAC

KAMAN SCIENCES CORPORATION

ATTN: DASIAC

SCIENCE APPLICATIONS INTL CORP

12 CY ATTN: CVR MR FARGO

DIRECTORY OF OTHER

DIVISION OF CONTINUING EDUCATION

ATTN: DR JOHN REDICK



Defense Threat Reduction Agency

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TDANP-TRC

August 1, 2001

MEMORANDUM TO DEFENSE TECHNICAL INFORMATION CENTER
ATTN: OCQ/MR LARRY DOWNING

SUBJECT: DOCUMENT CHANGES

The Defense Threat Reduction Agency Security Office reviewed the following documents in accordance with the Deputy Secretary of Defense Memorandum entitled, "Department of Defense Initiatives on Persian Gulf War Veterans' Illnesses" dated 22 March 1995, and determined that the documents were unclassified and cleared for public release:

DNA-TR-93-84, AD-B244408, Acoustic Resonance Spectroscopy in CW Verification Tooele Field Trial (August 1992).
DNA-TR-93-129-V1, AD-B192045, Global Proliferation – Dynamics, Acquisition Strategies and Responses, Volume 1 – Overview.
DNA-TR-93-129-V2, AD-B192046, Global Proliferation – Dynamics, Acquisition Strategies and Responses, Volume 2 – Nuclear Proliferation.
DNA-TR-91-216, AD-B163637, Harmonizing the Chemical Weapons Convention with the United States Constitution.
DNA-TR-92-180, AD-B175230, Evaluation of the Concept of a List for the BWC.
DNA-TR-92-61, AD-B167663, Basic State Party Functions and Skills Under CWC.
DNA-TR-92-66, AD-B167357, Domestic Reporting Requirements for Chemical Industry.
DNA-TR-91-213, AD-B163260, Analysis of the Interactions Between Treaties.
DNA-TR-93-70, AD-B177262, Chemical Weapons Convention Inspections of Private Facilities Application of United States Environmental and Safety Laws.
DNA-TR-92-182, AD-B173450, Commercial Products from Demilitarization Operations.
DNA-TR-91-217-V3, AD-B169350, Chemical Weapons Process Parameters, Volume 3 – Users' Guide.
DNA-TR-92-116-SUP, AD-B175292, Technical Ramifications of Inclusion of Toxins in the Chemical Weapons Convention (CWC), Supplement.
DNA-TR-92-128, AD-B175452, Task 1 Report Target Vapor Identification and Database Development.
DNA-TR-92-196, AD-B174940, Task 2 Report Algorithm Development and Performance Analysis.
DNA-TR-93-68, AD-B178109, CW Detection Instrument R&D Design Evaluation.

Enclosed is a copy of the referenced memorandum. If you have any questions, please call me at 703-325-1034.

Arndith Jarrett
ARDITH JARRETT
Chief, Technical Resource Center