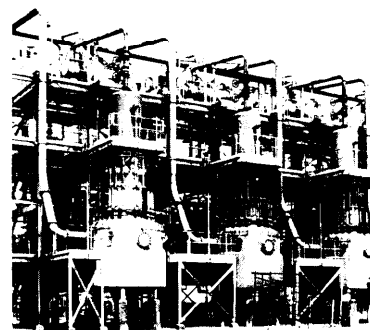


# Technical Aspects of Chemical Weapon Proliferation 2

Of the three categories of weapons of mass destruction, chemical weapons are the most likely to be used in warfare, and they remain a serious threat in regional conflicts despite the end of the Cold War. Although well-equipped troops can defend themselves against existing chemical agents with detectors, decontamination equipment, gas masks, and protective garments (albeit at a some cost in military effectiveness), chemical weapons can still have devastating effects when employed against defenseless civilians or poorly equipped (or unprepared) armies or guerrilla fighters. This fact was starkly demonstrated during the 1980s by Iraq's use of chemical weapons against Iran and its own Kurdish population.

The prospects for halting the proliferation of chemical weapons are mixed. On the one hand, several states are currently believed to possess or to be actively pursuing a chemical-warfare (CW) capability. On the other hand, the international community recently achieved a major step forward by concluding the Chemical Weapons Convention (CWC) after more than two decades of arduous negotiations. This treaty, which is expected to enter into force in January 1995, enacts a comprehensive global ban on the development, production, stockpiling, transfer to other countries, and use of CW agents and delivery systems. The CWC also provides for a highly intrusive verification regime that will provide a legal framework for enforcement. However, a number of key countries of concern have not yet signed the treaty.

This chapter describes the chief technical hurdles associated with the process of acquiring a militarily significant CW capability and discusses detectable signatures associated with each of these steps that might be used for monitoring or verification purposes. A separate report explains the tactical and strategic uses of chemical weapons, and the extent and



consequences of their spread.<sup>1</sup> The analysis here focuses on mustard and nerve agents because they are militarily the most effective and have been weaponized and stockpiled by several countries.

## SUMMARY

CW capabilities can vary greatly in sophistication. Although hundreds of tons of chemical agent would be required for large-scale use in a major conflict, smaller quantities could be effective for tactical engagements in regional wars or to terrorize population centers. An advanced CW capability would entail production of several agents with differing toxicities and physical characteristics, mated to different types of munitions, but a crude CW arsenal might contain only one or two agents and a simple delivery system such as an agricultural sprayer. The Iran-Iraq War of the 1980s saw the first protracted use of chemical weapons since World War I and the first use of nerve agents. According to Iranian sources, Iraqi chemical weapons accounted for some 50,000 Iranian casualties, including about 5,000 deaths.<sup>2</sup>

The growing availability of chemical know-how and production equipment, combined with the globalization of chemical trade, have given more than 100 countries the capability—if not necessarily the intent—to produce simple chemical weapons such as phosgene, hydrogen cyanide, and sulfur mustard. A smaller number have the capability to produce nerve agents such as GA (tabun), GB (sarin), GD (soman), and VX. The reason is that whereas mustard-gas production is very simple—particularly if thiodiglycol is available as a starting material—making nerve agents involves more complex and difficult reaction steps. Technical hurdles associated with the production process include the cyanation reaction for tabun and the alkylation reaction for the other

nerve agents. Alkylation requires high temperatures or highly corrosive reagents.

Chemical plants capable of manufacturing organic phosphorus pesticides or flame retardants could be converted over a period of weeks to the production of CW agents, although this would not be a simple process. Multipurpose plants would be easier to convert than single-purpose plants. The hurdles to acquiring a CW production capability are lower if a proliferant country seeks only to produce low-quality agent for immediate use and is willing to cut corners on agent shelf-life, safety, and environmental protection. Even so, CW agent production is still several steps removed from an operational CW capability, which also requires the design and development of effective munitions, the filling of the munitions before use, and mating with a suitable delivery system.

## | Indicators of CW Proliferation Activities

Many different types of precursor chemicals and equipment, many of them dual-use, are suitable for CW agent production. As a result, plant equipment or precursor chemicals per se do not provide a reliable means of distinguishing between legitimate and illicit production. Since most chemical facilities are relatively simple and multiuse, nonproliferation policies will need to focus on judgments of intent as well as capability.

Detection of CW proliferation—either within or outside the framework of an international treaty regime requires the correlation of multiple indicators and intelligence sources, ranging from satellites to human defectors. The probability of detecting a clandestine CW capability must therefore be evaluated in the context of the on-site inspection regime established by the Chemical Weapons Convention, as well as unilateral intelli-

<sup>1</sup>U.S. Congress, Office of Technology Assessment *Proliferation of Weapons of Mass Destruction: Assessing the Risks*, OTA-ISC-559 (Washington, DC: U.S. Government printing Office, August 1993).

<sup>2</sup>Mike Eisenstadt, *The Sword of the Arabs: Iraq's Strategic Weapons*, Washington Institute Policy Papers No. 21 (Washington DC: Washington Institute for Near East Policy, 1990), p. 6.

gence-gathering capabilities outside the treaty regime,

Specific indicators, or “signatures,” of CW acquisition activities may be detected through remote or on-site inspection of a suspect facility. Potential signatures include aspects of plant design and layout, testing of chemical munitions and delivery systems, presence of agents, precursors, or degradation products in samples from the production line or waste stream; and presence of “biomarkers” indicative of CW agent exposure in plant workers or in wild plants and animals living in the vicinity of a suspect facility. The utility of any given signature depends on the precise pathway taken by a given proliferant, including the choice of production process, the investment in emission-control technologies, and the amount of effort taken to mask or otherwise obscure the signature.

The production of both mustard and nerve agents results in long-lived chemical residues that can persist for weeks—in some cases years—after production has ceased. Such telltale chemicals can be detected in concentrations of a few parts per trillion with sensitive analytical techniques such as combined gas chromatography/mass spectrometry. For this reason, the ability to conceal illicit CW agent production in a known facility is probably limited, although a number of possible circumvention scenarios have been suggested. Existing analytical capabilities can be fully exploited, however, only if the inspectors are given intrusive access to a suspect site. Confidence in a country’s compliance may, therefore, diminish if such access is not forthcoming, or if the number of sites to be inspected is impractically large. Furthermore, because chemical analysis has the potential to yield “false positive” results when in fact no violation has occurred, chemical detection should not be seen as unequivocal evidence of CW production but rather as a key element in a broader array of indicators suggesting a violation.

While detection of CW production with near-site monitoring techniques (such as laser spect-

roscopy) appears promising, current technology cannot provide a high probability of detection—particularly for plants equipped with sophisticated emission-control systems. Nevertheless, rapid improvements in the sensitivity and specificity of analytical devices, combined with the rapid evolution of computer processing and data-storage technologies, promise to improve the utility of near-site monitoring in the not-too-distant future.

Finally, detection capabilities that are very impressive in certain circumstances maybe rather inconclusive in others—particularly if the proliferant is willing to expend time, effort, and resources to mask, obscure, or explain away his CW production activities. Thus, good detectability in principle does not necessarily mean high-confidence detection in practice. A robust inspection regime must comprise an interlocking web of inspections, all of which a cheater must pass in order to conceal his violations. Focusing inspections at a single point—even one believed to be a crucial chokepoint—would allow the cheater to focus his efforts on defeating the inspections.

### | Alternative Proliferation Pathways

Chemical-warfare agents can be produced through a wide variety of alternative routes. Just because the United States used a particular production pathway in the past does not mean that a proliferant country would not chose another route, although only relatively few are suited to large-scale production. For example, the United States and Iraq used different processes for the production of G-category nerve agents.

A proliferant country would either build a dedicated clandestine production facility or convert a commercial (single-purpose or multipurpose) chemical plant to CW agent production. In general, commercial pesticide plants lack the precursor materials, equipment, facilities, handling operations, and safety procedures required for nerve-agent production, and would therefore require weeks to months to convert. Binary

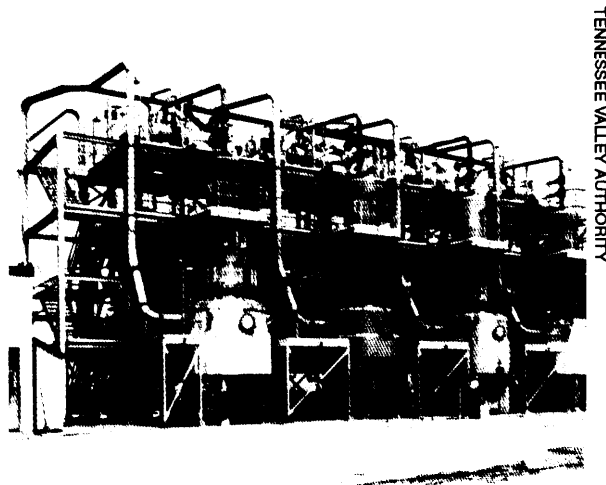
agents-consisting of two relatively nontoxic chemical components that when mixed together react to form a lethal agent-might be attractive to a proliferant because they are easier and safer to produce and handle, although they may be more complex to use in combat.

Each of the possible proliferation pathways involves tradeoffs among simplicity, speed, agent shelf-life, and visibility. The choice of pathway would therefore be affected by the urgency of a country's military requirement for a CW stockpile, its desire to keep the program secret, its level of concern over worker safety and environmental protection, and the existence of embargoes on precursor materials and production equipment.

### ACQUIRING A CW CAPABILITY

Although hundreds of thousands of toxic chemicals have been examined over the years for their military potential, only about 60 have been used in warfare or stockpiled in quantity as chemical weapons.<sup>3</sup> Physical properties required of CW agents include high toxicity, volatility or persistence (depending on the military mission), and stability during storage and dissemination. Lethal agents that have been produced and stockpiled in the past include *vesicants* such as sulfur mustard and lewisite, which bum and blister the skin, eyes, respiratory tract, and lungs; *choking agents* such as phosgene and chlorine, which irritate the eyes and respiratory tract; *blood agents* such as hydrogen cyanide, which starve the tissues of oxygen; and *nerve agents* such as sarin and VX, which interfere with the transmission of nerve impulses, causing convulsions and death by respiratory paralysis.

Unlike nuclear weapons, which require a large, specialized, and costly scientific-industrial base, CW agents can be made with commercial equipment generally available to



*Part of the U.S. Army's Phosphate Development Works, located on the grounds of Tennessee Valley Authority's National Fertilizer Development Center in Muscle Shoals, Alabama. Between 1953 and 1957, this facility met the Army's need for dichlor, a precursor needed to produce the nerve agent sarin.*

any country. Indeed, few military technologies have evolved *as little as* chemical weapons over the past half-century.<sup>4</sup> Current-generation mustard and nerve agents are based on scientific discoveries made during and between the two World Wars, and there have been few major innovations since then in either basic chemicals or manufacturing methods. The vast majority of the U.S. stockpile (in terms of tonnage) was produced during the 1950s and 1960s, when the United States managed to produce high-quality CW agents with what is now 30- to 40-year-old technology. Moreover, production techniques for the major CW agents have been published in the open patent or chemical literature, including data on reaction kinetics, catalysts, and operating parameters. According to one analyst, "The routes of production are generally known, and they can be pursued with relatively primitive

<sup>3</sup>World Health Organization@ *Health Aspects of Chemical and Biological Weapons: Report of a WHO Group of Consultants (Geneva: WHO, 1970)*, p. 23.

<sup>4</sup>In this **respect**, there is a significant difference between manmade chemical agents and biological toxins, whose production has been transformed by advances in biotechnology. See ch. 3.

equipment, especially by those who are not overly concerned with worker health and safety or environmental impacts.

As the commercial chemical industry has spread around the world in response to the urgent needs of developing countries for chemical fertilizers, pesticides, and pharmaceuticals, the availability of chemicals and equipment required to produce CW agents has increased. At the same time, thousands of applied organic chemists and chemical engineers from developing countries have been trained in related production technologies at universities in the United States, Europe, and the former Soviet Union.<sup>6</sup> According to Rear Adm. Thomas A. Brooks, former Director of Naval Intelligence:

The substantial pool of Western or Western-trained scientists, engineers and technicians has successfully been tapped for years by Third World states eager to acquire their expertise for missile development, nuclear, chemical and other weapon projects.<sup>7</sup>

The dual-use nature of many chemical technologies has made CW proliferation “an unfortunate side effect of a process that is otherwise beneficial and anyway impossible to stop: the diffusion of competence in chemistry and chemical technology from the rich to the poor parts of the world. Nevertheless, CW agent production is only one step on the path to acquiring a full capability to wage chemical warfare. A supertoxic agent, despite its lethality, does not become a usable weapon of war until it has been integrated with some form of munition or delivery system and made an integral component of a nation’s military planning and doctrine.

## | Acquisition Steps

The following steps are required for a proliferant country seeking to acquire a fully integrated CW capability (see figure 2-1):

1. acquire equipment and materials needed for CW agent production and the relevant expertise;
2. produce agents in small quantities at a pilot facility to work out technical details of the synthetic process, and then scale up to a production plant;
3. purchase suitable munitions and delivery systems (or design, prototype, test, and produce them indigenously);
4. fill the munitions with agent;
5. establish bunkers (or other storage facilities) and logistical support networks for the stockpiling, transport, handling, and use of bulk agents and munitions;
6. deliver chemical munitions to the military logistics system for storage and transport to the battle zone;
7. acquire individual and collective chemical defenses and decontamination equipment, and train troops how to fight in a chemical environment; and
8. develop strategic and tactical battle plans for CW use, and practice them in operational tests and field exercises.

To save time or money, a state seeking a more rudimentary CW capability might cut corners on some of these steps, for example, by omitting rigorous safety and waste-treatment measures during the production process. Proliferant states might also settle for a less robust logistical infrastructure than that developed by the United

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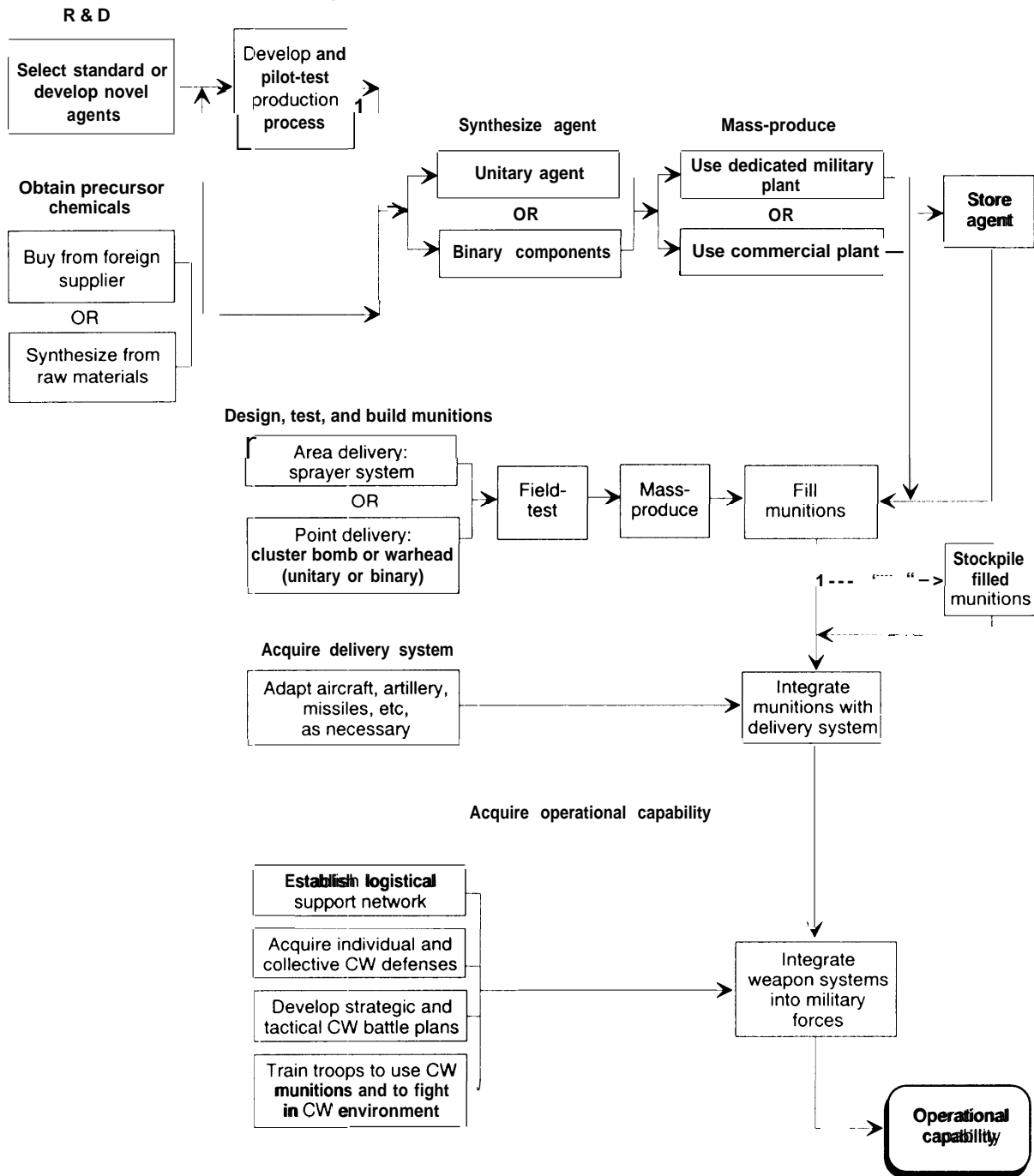
<sup>5</sup> Kyle Olson, “Disarmament and the Chemical Industry,” Brad Roberts, cd., *Chemical Disarmament and U.S. Security* (Boulder, CO: Westview Press, 1992), p. 100.

<sup>6</sup> In 1990, for example, foreigners accounted for a large fraction of full-time graduate students at U.S. universities studying chemistry (32 percent) and chemical engineering (42 percent). Commission on Professionals in Science and Technology, “Foreign Graduate Students” (table), *Scientific-Engineering-Technical Manpower Comments*, vol. 29, No. 6, September 1992, p. 13.

<sup>7</sup> Michael R. Gordon, “The Middle East’s Awful Arms Race: Greater Threats from Lesser Powers,” *The New York Times*, Apr. 8, 1990, sec. 4, p. 3.

<sup>8</sup> Julian Perry Robinson, “Chemical Weapons Proliferation: The Problem in Perspective,” Trevor Findlay, cd., *Chemical Weapons and Missile Proliferation* (Boulder, CO: Lynne Rienner, 1991), p. 26.

Figure 2-1—Chemical Weapon Acquisition



SOURCE: Office of Technology Assessment, 1993.

States and other nations with a broad, integrated military establishment. Finally, proliferants might forego protection and decontamination capabilities if the opponent lacks a CW capability and the losses resulting from “friendly fire” are considered an acceptable cost of war.<sup>9</sup>

## AGENTS AND PRODUCTION PROCESSES

### I Sulfur Mustard

Sulfur mustard (H), the main blistering agent used in warfare, is an oily liquid at room temperature that smells of garlic and ranges in color from clear to dark brown depending on purity.<sup>10</sup> It is readily absorbed by the skin and most clothing. Sulfur mustard is fairly persistent in the environment, presenting a hazard for days or even weeks depending on the weather. Compared with the more toxic nerve agents, sulfur mustard is relatively easy to produce and load into munitions, and it can be stockpiled for decades—especially when distilled—either as bulk agent or in weaponized form. The primary drawbacks of sulfur mustard as a CW agent are that:

- it must be used in relatively high concentrations to produce significant casualties;
- it freezes at a relatively high temperature—about 14 degrees Celsius (57 degrees Fahrenheit) for distilled mustard; and
- if not distilled to high purity, mustard tends to polymerize when stored for long periods, forming solids that precipitate out of solution and reduce the efficiency of dissemination.

Sulfur mustard has diffuse toxic effects on the body that may take as long as 3 hours or more to manifest themselves. The primary effect of an attack with sulfur mustard is to produce painful skin blistering and eye and lung irritation, resulting in a large number of wounded casualties who place an enormous burden on medical services. Heavy exposure to an aerosol of mustard or mustard vapor causes the lungs to fill with fluid, “drowning” the victim from within.<sup>11</sup> Nevertheless, only 2 to 3 percent of hospitalized American and British mustard casualties in World War I died, and a similar low death rate was reported for Iranian mustard casualties during the Iran-Iraq War.<sup>12</sup> Seven to 10 days after exposure, sulfur mustard can also cause a delayed impairment of immune function that increases vulnerability to bacterial infection and may lead to serious medical complications.

### PRODUCTION OF SULFUR MUSTARD

Nine production processes for sulfur mustard have been documented in the published chemical literature. During World War I, thousands of tons of mustard gas were produced from alcohol, bleaching powder, and sodium sulfite. During World War II, the two largest producers of mustard gas, the United States and the Soviet Union, used two common industrial chemicals—sulfur monochloride and ethylene—as starting materials.<sup>13</sup> A mustard-gas plant based on this method could be located at an oil refinery, which is an excellent source of ethylene and could also extract the necessary sulfur from petroleum or natural gas.<sup>14</sup>

<sup>9</sup> U.S. Congress, Office of Technology Assessment, *Who Goes There: Friend or Foe?*, OTA-ISC-537 (Washington DC: U.S. Government Printing Office, June 1993).

<sup>10</sup> Sulfur mustard may be produced in either crude form (H) or washed and vacuum-distilled (HD).

<sup>11</sup> Most of the Iranian fatalities caused by Iraqi use of sulfur mustard during the Iran-Iraq War were caused by liquid on clothing being inhaled over a long period in the hot desert sun. William C. Dee, U.S. Army Chemical-Biological Defense Command, personal communication 1993.

<sup>12</sup> Seth Schonwald, “Mustard Gas,” *The PSR Quarterly*, vol. 2, No. 2, June 1992, p. 93.

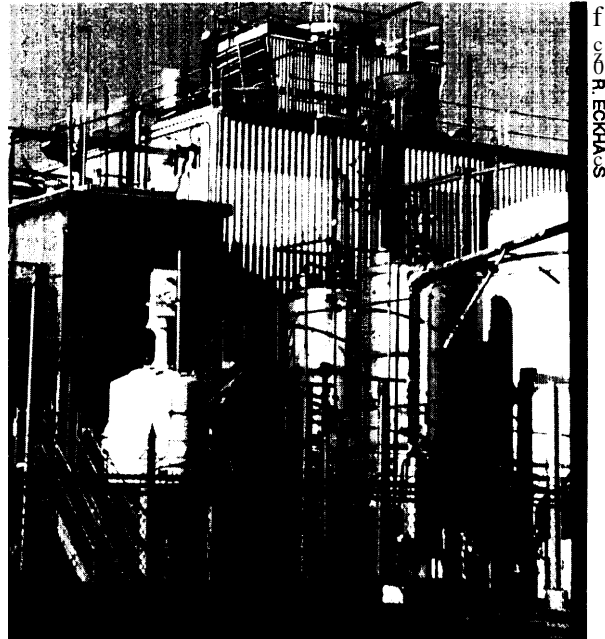
<sup>13</sup> Gordon Burck et al., *Chemical Weapons Process Parameters, Vol. I: Main Report* (Alexandria, VA: EAI Corp., Report No. DNA-TR-91-217-V1, November 1992).

<sup>14</sup> Gordon M. Burck and Charles C. Flowerree, *International Handbook on Chemical Weapons Proliferation* (New York, NY: Greenwood Press, 1991), p. 50.

## 22 | Technologies Underlying Weapons of Mass Destruction

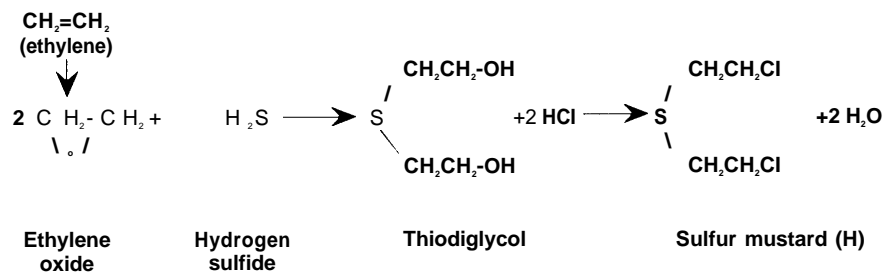
Today, the precursor of choice for any large-scale production of mustard gas is thiodiglycol, a sulfur-containing organic solvent that has commercial applications in the production of ballpoint pen inks, lubricant additives, plastics, and photographic developing solutions, and as a carrier for dyes in the textile industry. Thiodiglycol is just one step away from production of sulfur mustard, requiring only a reaction with a chlorinating agent like hydrochloric acid (HCl), a widely available industrial chemical.<sup>15</sup> (See figure 2-2.) Known as the Victor Meyer-Clarke Process, the chlorination of thiodiglycol was developed by Germany during World War I and adopted in the 1980s by Iraq. It does not require a particularly sophisticated chemical industry and could, indeed, be performed in a basement laboratory with the necessary safety precautions.

Sulfur mustard can be produced on an industrial scale on either a batch or continuous basis. Given the extreme corrosiveness of hot hydrochloric acid, it is advisable-but not essential-to



*Plant that produces the “dual-use” chemical thiodiglycol, which is both a key ingredient of ballpoint pen ink and an immediate precursor of mustard agent.*

**Figure 2-2—Production of Sulfur Mustard**



SOURCE: Stephen Black, “Vesicant Production Chemistry,” In Black, Benoit Morel, and Peter Zapf, *Technical Aspects of Verification of the Chemical Weapons Convention*, Internal Technical Report, Carnegie-Mellon Program on International Peace and Security, 1991, p. 56.

<sup>15</sup> See Ronald G. Sutherland, “Thiodiglycol,” in S. J. Lundin, ed., *Verification of Dual-Use Chemicals Under the Chemical Weapons Convention; The Case of Thiodiglycol*, SIPRI Chemical & Biological Warfare Series No. 13 (Oxford, England: Oxford University Press, 1991), pp. 24-30.



use corrosion-resistant reactors and pipes. This requirement might be reduced by substituting a less corrosive chlorinating agent for HCl or by replacing the production equipment as often as necessary. In order to improve the purity and stability of sulfur mustard in storage, corrosive byproducts can be removed by distillation or solvent extraction.

There are five U.S. producers of thiodiglycol and about eight foreign producers in five countries.<sup>16</sup> Most of these companies do not sell the chemical but use it internally in the manufacture of other products. In addition, about 100 firms worldwide purchase thiodiglycol for the synthesis of specialty chemicals and other industrial uses.<sup>17</sup> When Iraq began mustard-gas production in the early 1980s, it was unable to make thiodiglycol indigenously and ordered more than 1,000 tons from foreign sources.<sup>18</sup> In response to the threatened embargo on exports of thiodiglycol from Western countries, however, Iraq developed an indigenous production capability based on reacting ethylene oxide with hydrogen sulfide. Both of these ingredients are widely available. Hydrogen sulfide can be extracted from natural gas or crude oil, where it is often present as an impurity, or derived from elemental sulfur. Ethylene oxide is readily produced from ethylene, a major product of petroleum refining.

In sum, the production of mustard gas is relatively easy from a technical standpoint and could probably be concealed. While export controls on thiodiglycol might initially create a major hurdle for new proliferants, the effectiveness of controls will diminish as these countries

acquire an indigenous capability to produce it. Furthermore, just because synthesis from thiodiglycol is the ‘‘best’’ process does not mean that it will be used by a proliferant. Any of the other synthetic pathways could work just as well for a developing country and might be used to circumvent export controls.

## | Nerve Agents

Nerve agents are supertoxic compounds that produce convulsions and rapid death by inactivating an enzyme (acetylcholinesterase) that is essential for the normal transmission of nerve impulses. The nerve agents belong to the class of organophosphorus chemicals, which contain a phosphorus atom surrounded by four chemical groups, one of which is a double-bonded oxygen. Although many organophosphorus compounds are highly toxic, only a few have physical properties that give them military utility as nerve agents.<sup>19</sup> The difference in toxicity between pesticides and nerve agents derives from the nature of the chemical groups surrounding the phosphorus atom. In general, nerve agents are 100 to 1,000 times more poisonous than organophosphorus pesticides.<sup>20</sup>

Two classes of nerve agents, designated G and V agents, were produced in large quantities in the 1950s and '60s by the United States and the former Soviet Union. (See figure 2-3.)

The *G-series nerve agents* are known both by informal names and military code-names: tabun (GA), sarin (GB), GC, soman (GD), GE, and GF. This class of compounds was discovered in 1936 by Gerhard Schrader of the German firm IG

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<sup>16</sup> Leo ZefTel, personal communication, 1992; U.S. Department of Commerce, Bureau of Export Administration, Office of Foreign Availability, *Foreign Availability Review: 50 CW Precursor Chemicals (II)* (Washington, DC: Department of Commerce, Nov. 8, 1991), p. 54.

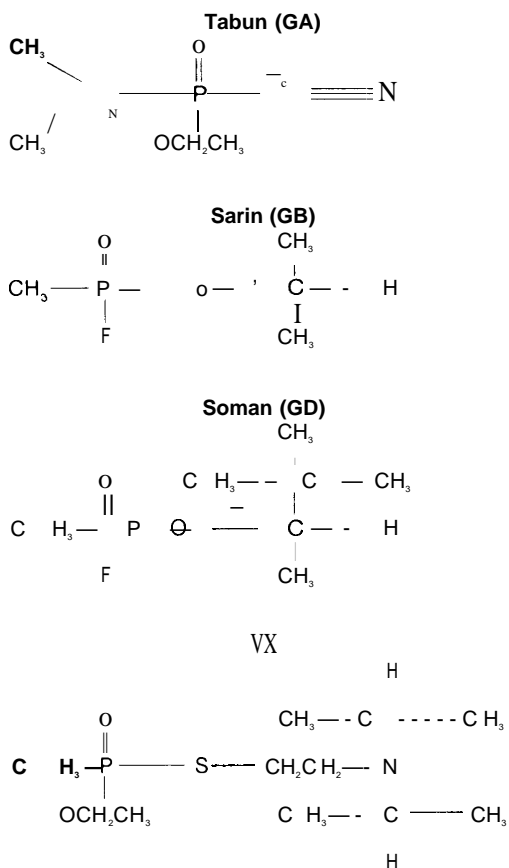
<sup>17</sup> Giovanni A. Snidle, ‘‘United States Efforts in Curbing Chemical Weapons Proliferation,’’ *World Military Expenditures and Arms Transfers 1989* (Washington, DC: U.S. Arms Control and Disarmament Agency, October 1990), p. 23.

<sup>18</sup> W. Seth Carus, *The Genie Unleashed: Iraq’s Chemical and Biological Weapons Program*, Policy Papers No. 14 (Washington, DC: The Washington Institute for Near East Policy, 1989), p. 13.

<sup>19</sup> Benjamin Witten, *The Search for Chemical Agents* (Aberdeen, MD: Edgewood Arsenal Special Technical Report, 1969).

<sup>20</sup> Alan R. Pittaway, ‘‘The Difficulty of Converting Pesticide Plants to CW Nerve Agent Manufacture,’’ Task IV, Technical Report No. 7 (Kansas City, MO: Midwest Research Institute, Feb. 20, 1970), p. 1.

Figure 2-3-Nerve Agents



SOURCE: JAYCOR, *Noncompliance Scenarios: Means By Which Parties to the Chemical Weapons Convention Might Cheat*, Technical Report No. DNA-TR-91-193, January 1993, pp. B-2-B-5.

Farben during research on new pesticides. Although tabun is relatively easy to produce, it is not as toxic as the other G agents. After World War II, details of German research on the G agents were published, and sarin and soman emerged as preferred agents for military purposes.

All the various G agents act rapidly and produce casualties by inhalation, although they also penetrate the skin or eyes at high doses

(particularly when evaporation is minimized and contact is prolonged by contamination of clothing).<sup>21</sup> Sarin evaporates faster than it penetrates the skin, but soman and GF are less volatile and pose more of a skin-contact hazard.

The *V-series nerve agents* include VE, VM, and VX, although only VX was weaponized by the United States. These agents were originally discovered in 1948 by British scientists engaged in research on new pesticides. Military development was then conducted by the United States and the Soviet Union, both of which began large-scale production of V agents in the 1960s.<sup>22</sup> VX is an oily liquid that may persist for weeks or longer in the environment. Although not volatile enough to pose a major inhalation hazard, it is readily absorbed through the skin. The lethal dose of VX on bare skin is about 10 milligrams for a 70 kilogram man.<sup>23</sup>

## PRODUCTION OF NERVE AGENTS

From the standpoint of production processes, the nerve agents can be clustered into three groups: tabun; sarin/soman, and VX.

### Tabun

The first militarized nerve agent and the simplest to produce, tabun (GA), is made from four precursor chemicals: phosphorus oxychloride ( $\text{POCl}_3$ ), sodium cyanide, dimethylamine, and ethyl alcohol. Most of these ingredients are widely available. Ethanol and sodium cyanide are commodity chemicals that are manufactured and sold in vast quantities; dimethylamine and phosphorus oxychloride are produced by companies in several countries for commercial applications in the production of pharmaceuticals, pesticides, missile fuels, and gasoline additives.

<sup>21</sup>Col. Michael A. Dunn and Frederick R. Sidell, "Progress in Medical Defense Against Nerve Agents," *Journal of the American Medical Association*, vol. 262, No. 5, Aug. 4, 1989, p. 649.

<sup>22</sup>Manuel Sanches et al., *Chemical Weapons Convention (CWC) Signatures Analysis* (Arlington, VA: System Planning Corp., Final Technical Report No. 1396, August 1991), p. 68.

<sup>23</sup>Witten, op. cit., footnote 19, pp. 92, 100.

The basic production process for tabun was developed by Germany during World War II and was later employed by Saddam Hussein's Iraq. Tabun synthesis does not require the use of corrosive starting materials and does not produce highly reactive intermediates. The two-step process involves mixing the ingredients and a carrier solvent in a reaction vessel equipped with a sodium-hydroxide scrubbing system to neutralize the gaseous hydrochloric acid (HCl) byproduct. A relatively simple air-tight enclosure is also needed to prevent the escape of toxic vapors. The ingredients must be added in the correct order, without heating, and the vessel cooled to keep the reaction from building up too much heat. Little or no distillation equipment is required, although the purity of tabun can be increased to more than 80 percent by removing the carrier solvent and the off-gasses by vacuum distillation.<sup>24</sup> In sum, tabun production is relatively easy because it does not include the difficult alkylation reaction needed to make the other nerve agents. The major technical hurdle in tabun synthesis is the cyanation reaction (in which a cyanide group is added to the central phosphorus), because of the difficulty of containing the toxic hydrogen cyanide HCN gas used as the reagent.

During World War II, Germany manufactured tabun in large quantities but never used it in combat. In early 1940, the Germans began construction of a huge tabun factory with the capacity to produce 3,000 tons of agent a month. Because of technical problems, however, it took the Germans over 2 years, until April 1942, to get

the plant operational.<sup>25</sup> The Iraqis also had difficulties with the manufacture of tabun, although they managed to produce a material with about 40 percent purity that was used in the Iran-Iraq War.<sup>26</sup>

#### Sarin/soman

Sarin (GB) and soman (GD) are both made in a batch process with the same basic reaction steps, but they contain different alcohol ingredients: isopropyl alcohol for sarin and pinacolyl alcohol for soman. (The choice of alcohol changes the toxicity and volatility of the product but does not affect the difficulty of production.) Phosphorus trichloride (PCl<sub>3</sub>) is the basic starting material for the synthesis of both agents and, depending on which of several alternative synthetic pathways is chosen, two to five steps are required to make the final product. The alternative syntheses all involve the same four reaction steps, which can be carried out in several different sequences.<sup>27</sup> During the 1950s, new production methods overcame the technical difficulties that had prevented the Germans from engaging in the large-scale production of sarin and soman during World War II. The introduction of these new methods enabled the U.S. sarin plant at Rocky Mountain Arsenal in Colorado to produce 10 tons of agent per day.

The synthesis of G agents entails three major technical hurdles. First, the production process involves the use of hot hydrochloric acid (HCl) and hydrogen fluoride (HF), both of which are extremely corrosive. The use of these compounds in reactors and pipes made of conventional steel

<sup>24</sup> S. Black, B. Morel, and P. Zapf, "Verification of the Chemical Convention" *Nature*, vol. 351, June 13, 1991, p. 516.

<sup>25</sup> Robert Harris and Jeremy Paxman, *A Higher Form of Killing: The Secret Story of Germ and Gas Warfare* (London: Chatto & Windus, 1982), pp. 55-56.

<sup>26</sup> Dee, op. cit., footnote 11.

<sup>27</sup> For example, the United States used the so-called "Di-Di" process to produce sarin. In this process, methylphosphonic dichloride (DC), or CH<sub>3</sub>POCl<sub>2</sub>, is partially reacted with hydrogen fluoride (HF) gas to make a roughly 50:50 mixture of DC and methylphosphonic difluoride (DF). The DC-DF mixture is then reacted with isopropyl alcohol. This reaction displaces chlorine atoms preferentially, resulting in the formation of sarin and hydrochloric acid (HCl) gas, which must be removed rapidly by distillation to avoid degradation of the nerve-agent product. (Pure DF is not used because the reaction with isopropyl alcohol would liberate HF gas, which is soluble in G agent and virtually impossible to degas, resulting in a highly corrosive mixture.)

results in corrosion measured in inches per year.<sup>28</sup> During World War II, the Germans lined their reactors with silver, which is resistant to HCl and HF. Today, corrosion-resistant reaction vessels and pipes are made of alloys containing 40 percent nickel, such as the commercial products Monel and Hastalloy.<sup>29</sup> Although it is possible to manufacture sarin and soman without corrosion-resistant reactors and pipes, the chance of major leaks is significantly increased compared with using corrosion-resistant equipment.

The second hurdle in the production of G agents is the alkylation reaction, in which a methyl group (-CH<sub>3</sub>) or an ethyl group (-CH<sub>2</sub>CH<sub>3</sub>) is added to the central phosphorus to form a P-C bond. This step is rarely used in the production of commercial pesticides and is technically difficult.

The third hurdle is that if high-purity agent with a long shelf-life is required, the supertoxic final product must be distilled—an extremely hazardous operation. Distillation is not necessary if a country plans to produce nerve agents for immediate use rather than stockpiling them. During the Iran-Iraq War, for example, Iraq gave priority to speed, volume, and low cost of production over agent quality and shelf-life. As a result, the sarin in Iraqi chemical munitions was only about 60 to 65 percent pure to begin with and contained large quantities of hydrogen fluoride (HF), both because of the synthesis process used and the deliberate omission of the distillation step. Although the Iraqis could have distilled their sarin to remove the excess HF, they chose not to do so because the batches of agent were intended to be used within a few days. To retard the rate of deterioration, sarin-filled shells were stored in

refrigerated igloos. Thus, whereas the distilled sarin produced by the United States in the early 1960s has retained a purity of more than 90 percent for three decades, the agent content of Iraqi sarin after 2 years of storage had generally degraded to less than 10 percent and in some cases below 1 percent.<sup>30</sup>

## VX

The persistent nerve agent VX has a phosphorus-methyl (P-CH<sub>3</sub>) bond and a phosphorus-sulfur bond but contains no fluorine. There are at least three practical routes to V-agents that might be used by proliferant countries. As with G agents, production of VX involves a difficult alkylation step.<sup>31</sup> Because the VX manufacturing process avoids the use of HF gas, however, it is less corrosive than the production of sarin and soman. Indeed, after the alkylation step has been completed, the rest of the synthesis is straightforward.

## PRODUCTION HURDLES

In summary, the technologies required for the production of mustard and nerve agents have been known for more than 40 years and are within the capabilities of any moderately advanced chemical or pharmaceutical industry. The technical hurdles associated with nerve-agent production are not fundamentally different from those associated with commercial products such as organophosphorus pesticides. The most technically challenging aspects include:

- the cyanation reaction for tabun, which involves the containment of a highly toxic gas;

<sup>28</sup> Stephen Black, Benoit Morel, and Peter Zapf, *Technical Aspects of the Chemical Weapons Convention: Interim Technical Report* (Pittsburgh, PA: Carnegie Mellon University, Program on International Peace and Security, 1991), p. 70.

<sup>29</sup> Although glass-lined reactors and pipes resist HCl corrosion, HF attacks glass and hence can only be used in metal reactors.

<sup>30</sup> United Nations Special Commission, "Second Report by the Executive Chairman of the Special Commission Established by the Secretary-General Pursuant to Paragraph 9 (b) (i) of Security Council Resolution 687 (1991)," UN Security Council document No. S/23268, Dec. 4, 1991, p. 5.

<sup>31</sup> The U.S. production method for VX, known as the Newport process, involved high-temperature methylation, in which phosphorus trichloride (PCl<sub>3</sub>) is reacted with methane gas (CH<sub>4</sub>) at a high temperature (500 degrees C) to form an alkylated intermediate, with a yield of only about 15 percent.

- the alkylation step for sarin, soman, and VX, which requires the use of high temperatures and results in corrosive and dangerous byproducts such as hot hydrochloric acid;
- careful temperature control, including cooling of the reactor vessel during heat-producing reactions, and heating to complete reactions or to remove unwanted byproducts;
- intermediates that react explosively with water, requiring the use of heat-exchangers based on fluids or oils rather than water; and
- a distillation step if high-purity agent is required.

While some steps in the production of nerve agents are difficult and hazardous, they would probably represent more of a nuisance than a true obstacle to a determined proliferant. The final distillation step can also be avoided if a proliferant country seeks to manufacture low-purity agent for immediate use and is prepared to cut corners on safety, environmental protection, and the life-span of the production equipment. Indeed, the United States produced nerve agents very effectively with 1950s technology and without the stringent safety and environmental standards that would be required today. In an attempt to conceal a CW production effort, a proliferant country might also resort to less-well-known production processes that had earlier been discarded because of their high cost, inefficiency, hazards, or need for unusual precursors or catalysts.

costs

A sulfur-mustard production plant with air-handling capabilities might cost between \$5 million and \$10 million to build. In contrast, a more sophisticated G-agent production facility

would cost between \$30 and \$50 million. Since waste-handling facilities would account for more than 50 percent of the cost of a G-agent plant, a “no-frills” production facility that did away with waste handling might cost about \$20 million.<sup>32</sup> Construction of a large-scale plant and equipment would be almost impossible for a developing country without outside assistance, but cost alone is unlikely to be the deciding factor for a determined proliferant.

### | Implications of New Technology

Given the well-understood production pathways of mustard and nerve agents and their record of use in warfare, a developing country that sought to acquire a CW capability would not need to develop and weaponize new agents. The development and production of novel CW agents would probably be undertaken only by nations with an advanced scientific-industrial base; even then, a major investment of time and resources would be required. During the 1930s, it took an advanced industrial country like Germany 6 years to put the first nerve agent, tabun, into production.<sup>33</sup>

Even so, the development of entirely new classes of CW agents remains a real possibility. In late 1992, a Russian chemist alleged that a military research institute in Moscow had developed a new binary nerve agent more potent than VX; he was subsequently arrested by the Russian Security Service for disclosing state secrets.<sup>34</sup> Another cause for concern is that some laboratories working on chemical defenses are studying the mode of action of nerve agents at the molecular level. Although the purpose of this research is to develop more effective antidotes, it could also assist in the development of novel

<sup>32</sup> Dee, op. cit., footnote 11.

<sup>33</sup> Gordon M. Burck, “Chemical Weapons Production Technology and the Conversion of Civil Production,” *Arms Control*, vol. 11, No. 2, September 1990, p. 145.

<sup>34</sup> “Mirzayanov, Fedorov Detail Russian CW Production,” *Novoye Vremya* (Moscow), No. 44, October 1992, pp. 4-9 (translated in FBIS-SOV-92-213, Nov. 3, 1992, pp. 2-7). See also, Will Englund, “Ex-Soviet Scientist Says Gorbachev’s Regime Created New Nerve Gas in ‘91,” *Baltimore Sun*, Sept. 16, 1992, p. 3.

compounds more deadly than existing nerve agents.<sup>35</sup> A more potent agent would not necessarily translate into greater military effectiveness, however, unless the dissemination system were improved as well.

Some experts are also concerned that even though the Chemical Weapons Convention (CWC) bans the development of *any* toxic chemical for warfare purposes, some countries might seek to circumvent the CWC verification regime by modifying existing agents to avoid detection or by weaponizing a “second string” of known but less effective poisons. The carbamates, for example, are a class of toxic pesticides that resemble organophosphorus nerve agents in that they inactivate the enzyme acetylcholinesterase. To date, the carbamates have not been developed as CW agents because they have a number of operational drawbacks: they are relatively unstable, are solids at room temperature (posing less of an inhalation threat), and are relatively easy to treat or pretreat with antidotes.<sup>36</sup> Even so, such chemicals might become more attractive as an alternative to standard nerve agents. Also of potential concern as novel CW agents are toxins of biological origin, which might be produced in militarily significant quantities with biotechnological techniques. Some toxins are thousands of times more potent than nerve agents, although they also have operational limitations. (See next chapter.)

Another potential threat is the use of penetrant chemicals to defeat chemical defenses, such as

“mask-breakers’ capable of saturating gas-mask filters made of activated charcoal.<sup>37</sup> Defensive equipment has long been modified to deal with certain small molecules of this type and is still being improved.<sup>38</sup> Although a variety of other means for penetrating masks and protective clothing have been examined over a period of many years, all of them have operational shortcomings.<sup>39</sup> Even so, penetrants remain a serious potential threat. If a new concept for penetrating CW defenses emerged that lacked the existing drawbacks, it could have a major impact on the overall military significance of chemical weapons. As one analyst has pointed out, the long-term danger is that “some future technological development might reverse the present ascendancy of the defense (i.e., antichemical protection) over the offense, thereby destroying a major incentive for deproliferation.”<sup>40</sup> Since such a technological breakthrough could trigger renewed competition in chemical weapons, measures to constrain research and development would be of major value in halting CW proliferation.

### | Precursor Chemicals for CW Agents

Chemicals that serve as starting materials in the synthesis of CW agents are known as ‘precursors. During the two world wars, the major powers produced CW agents from indigenous precursors. In World War I, for example, Germany manufactured chlorine and phosgene gas in huge volumes with existing chemical facilities.

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<sup>35</sup> External Affairs and International Trade <sup>cm@</sup> Verification Research Unit, *Verification Methods, Handling, and Assessment of Unusual Events in Relation to Allegations of the Use of Novel Chemical Warfare Agents* (Ottawa, Canada: External Affairs, March 1990), p. 10.

<sup>36</sup> There is, however, another side to the coin. The reversibility of carbamate poisoning (e.g., with atropine treatment) would offer an advantage to the attacker in the case of accidental leaks or spills. Similarly, the relative instability of carbamates would result in less persistence in the environment, facilitating occupation of attacked territory.

<sup>37</sup> Walter J. Stoessel, Jr., chairman, et al., *Report of the Chemical Warfare Review Commission* (Washington, DC: U.S. Government Printing Office, June 1985), p. 32.

<sup>38</sup> Telephone interview with Tom Dashiell, consultant, U.S. Arms Control and Disarmament Agency, Feb. 9, 1993.

<sup>39</sup> J. Perry Robinson, ed., *The Chemical Industry and the Projected Chemical Weapons Convention*, Vol. Z: *Proceedings of a SIPRI/Pugwash Conference* (New York, NY: Oxford University Press, 1986), p. 70.

<sup>40</sup> Julian Perry Robinson, “The Supply-Side Control of the Spread of Chemical Weapons,” Jean-Francois Rioux, cd., *Limiting the Proliferation of Weapons: The Role of Supply-Side Strategies* (Ottawa, Canada: Carleton University Press, 1992), p. 70.

Table 2-1—Dual-Use Chemicals

Dual-use chemical	CW agent	Commercial product
Thiodiglycol	Sulfur mustard	Plastics, dyes, inks
Thionyl chloride	Sulfur mustard	Pesticides
Sodium sulfide	Sulfur mustard	Paper
Phosphorus oxychloride	Tabun	Insecticides
Dimethylamine	Tabun	Detergents
Sodium cyanide	Tabun	Dyes, pigments, gold recovery
Dimethyl methylphosphonate	G Agents	Fire retardants
Dimethyl hydrochloride	G Agents	Pharmaceuticals
Potassium bifluoride	G Agents	Ceramics
Diethyl phosphite	G Agents	Paint solvent

SOURCE: Giovanni A. Snidle, "United States Efforts in Curbing Chemical Weapons Proliferation," U.S. Arms Control and Disarmament Agency, *World Military Expenditures and Arms Transfers 7939* (Washington, DC: U.S. Government Printing Office, October 1990), p. 23.

Today, however, the globalization of the chemical industry has led to large international flows of dual-use chemicals.

Many of the basic feedstock chemicals used in the production of nerve agents (e.g., ammonia, ethanol, isopropanol, sodium cyanide, yellow phosphorus, sulfur monochloride, hydrogen fluoride, and sulfur) are commodity chemicals that are used in commercial industry at the level of millions of tons per year and hence are impossible to control. Monitoring their sale would also be of little intelligence value because the imprecision of international-trade data would make it impractical to detect the diversion of militarily significant quantities. Hydrogen fluoride, for example, is used at many oil refineries and can be purchased commercially in large quantities; it is also easily derived from phosphate deposits, which usually contain fluorides.

Most of the key precursors for nerve-agent production also have legitimate industrial uses, but the fact that they are manufactured in much smaller volumes makes them somewhat

easier to control. These chemicals include phosphorus trichloride (with 40 producers worldwide), trimethyl phosphite (21 producers), and—for tabun only—phosphorus oxychloride (40 producers).<sup>41</sup> Phosphorus oxychloride, for example, is used extensively in commercial products such as hydraulic fluids, insecticides, flame retardants, plastics, and silicon. Similarly, dimethyl methylphosphonate (DMMP), an intermediate in nerve-agent production, is produced as a flame retardant by 11 companies in the United States and 3 in Europe (Belgium, United Kingdom, and Switzerland).<sup>42</sup> (See table 2-1.)

Developing countries seeking a CW capability generally lack the ability to manufacture key precursor chemicals and must purchase them from foreign sources, typically at well above normal market rates. Because of this dependency, Western governments have attempted to slow CW proliferation by establishing a committee known as the Australia Group, which coordinates national export-control regulations to restrict the sale of key CW precursors to sus-

41 U.S. Department of Commerce, op. cit., footnote 16, pp. 39, 58, 36, respectively.

42 Ibid., pp. 22-23. Lists of sources of CW precursors vary, since some lists include only those companies with an annual production volume greater than 4,500 kilograms or 5,000 pounds.

pected proliferants.<sup>43</sup> Nevertheless, the export controls coordinated by the Australia Group cannot prevent countries that are outside this body from selling precursor chemicals. Indeed, as Western countries have tightened CW-related controls, exports from developing nations such as India have increased. Of the 54 precursor chemicals whose exports are regulated by the Australia Group countries, Indian companies export about 15, only 4 of which are subject to Indian government export controls.<sup>44</sup>

Furthermore, to the extent that immediate precursors for mustard and nerve agents are controlled by the Australia Group, a proliferant might seek to circumvent such export controls in the following ways:

- Substituting an uncontrolled precursor chemical for one that is controlled. For example, although thionyl chloride is subject to export controls as a chlorinating agent for producing nerve agents, a proliferant could easily substitute some other chlorinating agent (e.g., phosgene, sulphuryl chloride) that is not on any export-control list. Thus, the technical means may exist to bypass any particular technology-transfer barrier.<sup>45</sup>
- Purchasing relatively small amounts of the same or different precursor chemicals from multiple sources, instead of obtaining large quantities from a single source. For example, a country might purchase several different types of chlorinating agent for the conversion of thiodiglycol to sulfur mustard. Such a purchasing strategy would reduce the visibility of CW production, although it would also increase the complexity of the production process.
- Producing more obscure (but still effective) CW agents whose precursors are still available. For example, production of the nerve agent soman (GD) requires pinacolyl alcohol, which has no commercial uses and whose export is restricted by the Australia Group. Because of this embargo, Iraqi military chemists chose instead to produce a 60:40 mixture of sax-in and GF (a less common nerve agent of intermediate persistence).<sup>46</sup> Sarin is made with isopropyl alcohol (ordinary rubbing alcohol), while GF is made with cyclohexyl alcohol (an industrial decreasing agent). Unlike pinacolyl alcohol, both isopropyl alcohol and cyclohexyl alcohol are common industrial chemicals that are not subject to export controls.
- “Back-integrating,” or acquiring an indigenous capability to manufacture precursor chemicals from simpler compounds whose export is not controlled or that are available from domestic sources. For example, thiodiglycol, the immediate precursor of sulfur mustard, can be produced in a batch process by reacting ethylene oxide with hydrogen sulfide. Both of these ingredients can be derived from oil or natural gas. Before the Persian Gulf War, Iraq built a huge production line at its Basra petrochemicals complex that was capable of manufacturing 110,000 tons of ethylene per year.<sup>47</sup>

In the case of nerve agents, all of the key precursors can be made from the most basic starting materials; including phosphorus, chlorine, and fluorine. The production facilities needed to make these precursors from raw materials are not particularly large and could be

<sup>43</sup> Julian Perry Robinson, “The Australia Group: A Description and Assessment,” Hans Guenter Brauch et al., eds., *Controlling the Development and Spread of Military Technology* (Amsterdam: W University Press, 1992), pp. 157-176.

<sup>44</sup> Michael R. Gordon, “U.S. Accuses India on Chemical Arms,” *New York Times*, Sept. 21, 1992, p. A7.

<sup>45</sup> Robinson, “The Supply-Side Control of the Spread of Chemical Weapons,” *op. cit.*, footnote 40, p. 68.

<sup>46</sup> U.S. Department of Defense, *The Conduct of the Persian Gulf War: Final Report to Congress* (Washington, DC: Department of Defense, April 1992), p. 18.

<sup>47</sup> Kenneth R. Timmerman, *The Death Lobby: How the West Armed Iraq* (Boston: Houghton-Mifflin, 1991), p. 35.



embedded in an existing industrial complex, although large amounts of energy would be required. During the Iran-Iraq War, for example, the Australia Group made a concerted effort to prevent Iraq from obtaining supplies of phosphorus oxychloride ( $\text{POCl}_3$ ), a key precursor of tabun. In response, Baghdad built a plant to manufacture phosphorus oxychloride indigenously, using raw phosphate ore from its huge phosphate mine at Akashat, so that it was no longer vulnerable to supplier embargoes of this precursor.<sup>48</sup>

Iraq also tried to apply back-integration to sarin production. In 1988, the Iraqi government contracted two West German companies to build three chemical plants at Al Fallujah, 60 miles west of Baghdad, for the conversion of elemental phosphorus and chlorine into phosphorus trichloride ( $\text{PCl}_3$ ), a key sarin precursor.<sup>49</sup> Baghdad also planned to produce hydrogen fluoride (HF), another essential ingredient in sarin production, by extracting it from phosphate ore with sulfuric acid. By the time of the August 1990 invasion of Kuwait, Iraq was on its way to building an indigenous capability to produce all of the major precursors of tabun and sarin, although it ultimately did not achieve this objective.<sup>50</sup>

The Iraqi case suggests that a country with large deposits of phosphate ore, a well-developed petrochemical industry, plentiful energy supplies, and access to the necessary technical know-how can develop an indigenous capability to produce all the major precursors of mustard and nerve agents. This ‘back-integration’ strategy would enable such a country to circumvent any foreign export-control regime designed to deny it access to CW agent precursors. Nevertheless, developing a back-integration capability is a large and costly undertaking, and may therefore be

beyond the means of all but the richest and most ambitious states of the developing world.

### | Containment and Waste Treatment

Because of the toxicity of CW agents, containment measures may be taken to ensure the safety of the plant workers and the nearby population. Such measures include air-quality detectors and alarms, special ventilation and air-scrubbing systems, protective suits and masks, and chemical showers for rapid decontamination. The safety and ventilation measures at the Iraq’s Al Muthanna CW production plant included measures comparable to U.S. procedures in the 1960s, when most of the U.S. chemical weapon stockpile was produced.<sup>51</sup>

For this reason, one should not use current U.S. safety and environmental standards as the norm when judging the likely proliferation paths of developing countries. If a ruthless government is willing to tolerate large numbers of injuries or deaths among production workers, CW agents can be manufactured in a very rudimentary facility with few, if any, systems in place to protect worker safety or the environment. In the former Soviet Union, for example, closed CW-agent production facilities were only introduced in the 1950s; before then the production process was entirely open to the atmosphere. According to a Russian scientist, production of blister agents during World War II took place under horrifying conditions:

In Chapayevsk we sent many thousands of people “through the mill” during the war. Soldiers who had been deemed unfit worked at the plant. Production was completely open: mustard gas and lewisite were poured into shells from kettles and scoops! In the space of a few months the “workers in the rear” became invalids and died. New people were brought into

<sup>48</sup> Ibid., p. 52.

<sup>49</sup> Carus, *op. cit.*, footnote 18, pp. 22-27.

<sup>50</sup> “News Chronology: August through November 1991,” *Chemical Weapons Convention Bulletin*, No. 14, December 1991, p. 8.

<sup>51</sup> Dee, *op. cit.*, footnote II.

production. Once during the war a train bringing reinforcements was delayed for some reason, and the plant stopped work. There was simply no one there to work! In nearby villages and hamlets there is probably no family which has not had a relative die in chemical production.<sup>52</sup>

If a proliferant country is concerned about protecting its environment or population (or wishes to cover up telltale evidence of its CW activities), the treatment and disposal of wastes from CW agent production poses a technical challenge. The waste stream contains hot acids contaminated with lethal agents and a large quantity of phosphates. Cleaning out the production line also requires large quantities of decontamination fluid, which becomes mixed with chemical agent and must be chemically or thermally destroyed to dispose of it in an environmentally sound manner. In the most modern plants, many spent or unused chemicals (e.g., DMMP, thionyl chloride) are recycled back into the production process. With the effective use of recycling, about one-half ton to 1 ton of waste is generated for each ton of nerve agent produced; without recycling, the ratio of waste to product is much higher.<sup>53</sup>

### | Weaponization of CW Agents

The weaponization of CW agents involves three steps:

1. the use of chemical additives to stabilize or augment the effects of a CW agent;
2. the design and production of munitions for dispersal of agent; and
3. the filling, storage, and transport of munitions.

Each of these steps is discussed in detail below.

### CHEMICAL ADDITIVES

The principal military requirements of a CW agent are that it be sufficiently toxic to produce large numbers of casualties, and thermally and mechanically stable enough so that it can survive explosive dissemination or passage through a spray device. Several chemicals may be added to CW agents to allow long-term storage or to enhance their military effects against personnel:

- *Stabilizers (e.g., amines)* prevent the degradation of CW agents exposed to hot temperatures or stored for long periods by absorbing acids released by chemical decomposition. Although CW agents filled into munitions do not require a long shelf-life if they are used immediately in combat, stockpiled munitions require stabilizers to prevent deterioration over a period of years.
- *Freezing-point depressants* lower the freezing point of liquid CW agents (primarily mustard) to permit use under winter conditions.
- *Thickeners* increase the viscosity and persistence of liquid agents.
- *Carriers* increase the airborne concentration of an agent like sulfur mustard, which is not very volatile at normal temperatures. During World War II, Germany did research on the potential use of silica powder as a potential carrier for mustard agents. A large quantity of sulfur mustard can be absorbed onto the powder and dispersed as a dust cloud. Because it contains a higher concentration of agent, “dusty mustard” produces more serious and rapid casualties than droplets of liquid agent.<sup>54</sup>

<sup>52</sup>“Mirzayanov, Fedorov Detail Russian CW production” op. cit., footnote 34, p. 4.

<sup>53</sup>Crawford & Russell, Inc., *Selection and Demonstration of the Most Suitable Processor for the Production of Methylphosphonic Dichloride* (Aberdeen Proving Ground, MD: U.S. Army Chemical Research, Development and Engineering Center, Dec. No. CRDEC-CR-87086, June 1987).

<sup>54</sup>J. Perry Robinson and Ralf Trapp, “Synthesis and Chemistry of Mustard Gas,” S.J. Lundin, ed., *Verification of Dual-use Chemicals under the Chemical Weapons Convention: The Case of Thiodiglycol*, SIPRI Chemical & Biological Warfare Studies No. 13 (New York Oxford University Press, 1991), p. 8.

- *Antiagglomerants*, such as colloidal silica prevent caking of powdered agent.

Although stabilizers are added routinely to CW agents, thickeners and carriers are more difficult to use. Thickeners, for example, do not readily go into solution and may take several hours to dissolve. Countries that do not require agents with high effectiveness or a long shelf-life may simply choose not to use additives, thereby simplifying the production process.

### FILLING OPERATIONS

In a CW agent production facility, the toxic material may flow directly from the production reactors to a munitions filling plant, where it is loaded into artillery shells, rockets, bombs, or spray tanks. Alternatively, the agent may be stored in bulk so that military missions can be considered when matching agents to munitions, or in the expectation that new delivery systems will be developed.

Because the filling operation is extremely hazardous, it is typically performed inside a sealed building with a controlled atmosphere; the filling machines themselves are totally enclosed and sealed from the external environment. The primary technical challenge is to seal the **super-toxic** liquid inside the munition without leakage and then to decontaminate the external surfaces. Iraq filled its unitary CW munitions on an enclosed, automated assembly line at the Al Muthanna production complex near Samarra. Such filling and sealing operations typically take about 2 to 3 minutes per projectile.<sup>55</sup>

A proliferant country might also fill CW munitions manually, although this operation would be labor-intensive and extremely dangerous. (Recall the quote above describing the manual filling of shells at a Soviet mustard plant during World War II.) During manual filling, a plant worker wearing a gas mask and protective clothing

transfers the agent through a hose from a storage vessel to the munition, which must then be plugged and sealed without any vapor loss or spillage. In wartime, filled munitions would be transported from stockpiles to positions on the battlefield from which they would be used. Other preparatory activities, such as inserting fuses and bursters, would also be required.

### MUNITIONS DESIGN

**Chemical** munitions are designed to convert a bulk payload of liquid or powdered agent into an *aerosol* of microscopic droplets or particles that can be readily absorbed by the lungs, or a *spray* of relatively large droplets that can be absorbed by the skin.<sup>56</sup> An aerosol consists of droplets between 1 and 7 microns (thousandths of a millimeter) in diameter, which remain suspended in the air for several hours and are readily inhaled deep into the lungs. In contrast, a spray capable of wetting and penetrating the skin consists of droplets at least 70 microns in diameter.<sup>57</sup>

A volatile agent like sarin is disseminated as a fine aerosol to create a short-term respiratory hazard. More persistent agents like sulfur mustard and VX are dispersed either as an aerosol (for respiratory attack) or as a coarse mist (for skin attack or ground contamination). After dissemination, nonvolatile agents may remain in puddles on the ground for weeks at a time, evaporating very slowly. The quantity of agent required to accomplish a particular military objective depends on the toxicity of the material involved and the efficiency of dissemination.

Many of the design specifications for chemical munitions can be found in the open patent literature, and suitable munitions production plants exist in many parts of the world. An aerosol or spray of CW agent may be disseminated by explosive, thermal, pneumatic, or mechanical means. The simplest device for deliver-

<sup>55</sup>Dee, op. cit., footnote 11.

<sup>56</sup>J. H. Rothschild, *Tomorrow's Weapons: Chemical and Biological* (New York, NY: McGraw-Hill, 1964), p. 66.

<sup>57</sup>Edward M. Spiers, *Chemical Weaponry: A Continuing Challenge* (New York, NY: St. Martin's Press, 1989), p. 21.

ing CW agents is a liquid spray tank mounted on an airplane or helicopter; such systems are commercially available for the dissemination of agricultural chemicals. To deliver an aerosol or spray of agent close enough to the ground to produce casualties, however, an aircraft must fly at low altitude and is thus vulnerable to air defenses, if they exist.

CW agents can also be delivered with a wide variety of munitions. During the Iran-Iraq War, for example, Iraq delivered mustard and tabun with artillery shells, aerial bombs, missiles, rockets, grenades, and bursting smoke munitions.<sup>58</sup> A bursting-type munition is packed with chemical agent and a high-explosive burster, a fuse, and a detonator; the use of more explosive produces a freer aerosol but may destroy much of the agent in the process. The fuse may be designed either to explode on impact with the ground or, using a proximity fuze, at an altitude of about 15 feet to enhance the formation of the aerosol cloud.<sup>59</sup> Sarin does not burn, but VX does and is therefore disseminated nonexplosively from a spray tank or by simple injection into the air stream from an aircraft or glide bomb.

#### Binary munitions

Chemical munitions can be either *unitary* or *binary in* design. Unitary munitions are filled with CW agent at a loading facility (often colocated with the production plant) before being stored and transported, so that only a fuse need be added before use. Binary munitions, in contrast, contain two separate canisters filled with relatively nontoxic precursor chemicals that must

react to produce a lethal agent. The two components are either mixed together manually immediately before firing or are brought together automatically while the binary bomb or shell is in flight to the target. Contrary to general belief, the chemicals produced in binary weapons are not novel CW agents but rather well-known ones, such as sarin, soman, and VX. (For technical reasons, tabun cannot be produced in a binary system).

The United States developed three binary chemical munitions: a 155mm artillery shell to deliver sarin against enemy troop concentrations on the battlefield; the BIGEYE spray bomb to deliver VX against fixed targets deep behind enemy lines; and a warhead for the Multiple Launch Rocket System (MLRS) containing a mixture of intermediate-volatility agents.<sup>60</sup> The binary artillery shell is a liquid/liquid system: one of the two precursor chemicals is isopropyl alcohol (rubbing alcohol), while the other, methylphosphonic difluoride (DF), is less toxic than tear gas.<sup>61</sup> In contrast, the BIGEYE spray bomb is a solid/liquid system: after the bomb is released, a pyrotechnic **gas** cartridge mixes particulate sulfur with a liquid precursor code-named QL to form VX. After the reaction has occurred, the bomb glides across the target, dispersing VX in its wake as a spray.<sup>62</sup> The development of advanced binary munitions entails considerable engineering challenges, both to accommodate the two components in a ballistically sound package and to effect the necessary chemical reaction during the flight of the shell or bomb.

<sup>58</sup> Harvey J. MacGeorge, "Iraq's Secret Arsenal," *Defense & Foreign Affairs*, January/February 1991, p. 7; MacGeorge, "The Growing Trend Toward Chemical and Biological Weapons Capability," *Defense and Foreign Affairs*, April 1991, p. 6.

<sup>59</sup> Burck and Flowerree, *op. cit.*, footnote 14, p. 506-507.

<sup>60</sup> Dan Boyle, "An End to Chemical Weapons: What Are the Chances?" *International Defense Review*, vol. 21, September 1988, p. 1088. Although the 155mm shell and the BIGEYE bomb were produced, the MLRS system was terminated in the final stages of development.

<sup>61</sup> In air, DF has an LD<sub>50</sub> (lethal dose in 50 percent of a population) of 67,000 mg/min/m<sup>3</sup>, compared with 63,000 mg/min/m<sup>3</sup> for CS (tear gas). DF and isopropyl alcohol are loaded into the munition in separate canisters; when the round is fired, the forces of acceleration rupture the wall between the canisters and allow the two reagents to mix. By the time the shell strikes the earth, the reaction is complete; the fuze detonates a burster charge, which disseminates a cloud of aerosolized sarin. Dee, *op. cit.*, footnote 11.

<sup>62</sup> Dee, *op. cit.*, footnote 1.

Nevertheless, binary CW weapons do not necessarily require sophisticated munition designs, since the two precursor chemicals can be premixed manually on the ground immediately prior to use. Iraq, for example, developed crude binary nerve-agent bombs and missile warheads because its DF precursor was very impure, causing the sarin product to decompose rapidly. As a result, the Iraqis planned to mix the binary precursors at the last possible moment before firing. At the Al Muthanna CW production facility, Iraqi workers half-filled 250-kilogram aerial bombs and Scud missile warheads with a mixture of isopropyl and cyclohexyl alcohols, and stored the DF component separately in plastic jerry cans. The operational plan was that just before the bombs and missiles were prepared for launch, a soldier wearing a gas mask would open a plug in the bomb casing or warhead and pour in the contents of four jerry cans of DF; the ensuing reaction would result in a 60-40 mixture of sarin and GF (a more persistent nerve agent).<sup>63</sup>

Binary weapons offer advantages in terms of ease and safety of production, storage, and transport, and hence might be attractive to potential proliferants. Nevertheless, binary weapons create operational drawbacks on the battlefield. The two precursors must either be premixed by hand—a dangerous operation—or separate canisters containing the two ingredients must be placed inside each munition immediately before firing.

#### Cluster bombs

One way to increase the area coverage of an aerial bomb or missile warhead is by means of cluster munitions, in which the chemical payload is broken up into many small bomblets (submunitions) that are released at altitude and scatter over a large “footprint” on the ground. During World War II, the United States developed chemical cluster bombs that carried 100 bomblets, each containing 5 kg of mustard. Such weapons were



UNITED NATIONS, H. ARVIDSSON

*United Nations inspector sampling DF, a nerve agent precursor that had been dumped into a pit in Iraq. The Iraqis stored DF separately from their chemical munitions, intending to add it just before use to form the nerve agents sarin and GF.*

designed to release the bomblets in a random pattern at an altitude of 1,000 feet; individual parachutes slowed the descent of the bomblets so they would not bury themselves in the ground.

#### MISSILE DELIVERY SYSTEMS

Ballistic missile systems such as the Soviet-designed Scud-B (with a range of 300 km) and FROG-7 (with a range of 67 km) can deliver warheads bulk-filled with chemical agent. Iraq developed bulk chemical warheads for its Al-Hussein modified Scud missiles (with an extended range of 500 to 600 km), although there is

<sup>63</sup> Terry J. Gander, “Iraq—The Chemical Arsenal,” *June’s Intelligence Review*, vol. 4, No. 9, September 1992, p. 414.

UNITED NATIONS, SHANKAR KUNHAMBU



*Iraqi worker preparing to open up for inspection a chemical warhead developed for Iraq's modified Scud missile.*

no evidence that they were actually tested.<sup>64</sup> United Nations inspectors in Iraq were shown 30 CW missile warheads, of which 16 had unitary sarin warheads. The other 14 were of the “binary” type and were partially filled with a mixture of alcohols pending the addition of DF stored in jerry cans nearby.

During the 1950s, the United States also developed CW cluster warheads for a series of rockets and missiles, including the Honest John, Sergeant, Improved Honest John, and the developmental LANCE warhead. A cluster warhead, however, cannot cover an area large enough to ensure that a missile as inaccurate as a Scud will deliver chemical agent to a particular target. Moreover, the area covered by a cluster warhead is somewhat unpredictable, since it depends to a large extent on the terrain and weather in the target zone.

Cruise missiles and remotely piloted vehicles (RPVs) are also potential CW delivery systems. During World War II, the Germans considered

filling the warhead of the V-1 flying bomb with phosgene gas instead of the normal 800 kilograms of high explosive. They decided against this proposal, however, after calculating that high explosives would actually produce more casualties than gas.<sup>65</sup> Nevertheless, a cruise missile or long-range RPV fitted with a 500 kg spray tank would be a cheap and effective delivery system that could lay down a linear spray cloud of CW agent.

In sum, systems suitable for delivering CW agents are widely available, and even the development and production of crude (manually mixed) binary weapons does not require a sophisticated industrial base. These observations suggest that the weaponization step does not pose a major technical bottleneck to the acquisition of a CW capability.

## INDICATORS OF CW PROLIFERATION ACTIVITIES

Verification of the international ban on chemical weapons will require the capability to detect militarily significant production of CW agents in a timely manner. Even a small production facility could manufacture militarily significant quantities of CW agent if it is operated for several years. Over a decade, a pilot-scale plant producing 10 tons of agent per year would accumulate 100 tons—a militarily significant quantity under certain contingencies.<sup>66</sup> Such long-term accumulation would, however, require distilling the agent to ensure a long shelf-life, thereby increasing complexity and cost; otherwise the total quantity of agent would be reduced by deterioration over the 10-year period. Increasing the number or scale of the production plants would reduce the length of time needed to accumulate a militarily significant stockpile.

<sup>64</sup> Declan McHugh, “~w Conference,” *Trust and Verify*, No. 35, January 1992, p. 2.

<sup>65</sup> Harris and Paxman, op. cit., footnote 25, p. 59.

<sup>66</sup> Manuel L. Sanches et al., *Analysis of Signatures Associated with Noncompliance Scenarios*, Report No. DNA-TR-92-74 (Arlington, VA: System Planning Corp., January 19'93), p. 7.

Monitoring measures designed to detect illicit CW production may be *cooperative*, within the framework of the Chemical Weapons Convention (CWC), or *noncooperative*, based on intelligence agents, remote sensing, and covertly placed monitoring devices that are not part of a negotiated regime. The cooperative monitoring regime established by the CWC requires participating countries to submit declarations, which will then be checked through “routine” onsite inspections; discrepancies may suggest illicit activities. To deter clandestine CW agent production, the treaty also provides for “challenge” inspections at government or private facilities, declared or undeclared. The advantage of the cooperative regime is that it permits direct access to production facilities, albeit in a tightly circumscribed manner. In contrast, unilateral intelligence-gathering efforts have the advantage that they are not constrained by agreed restrictions on data collection. The two approaches are not mutually exclusive and can be employed in a complementary manner.

Several potential indicators, or “signatures,” of CW development, production, and weaponization are discussed below. Although each signature taken in isolation is probably inadequate to prove the case, a “package” of signatures from various sources may be highly suggestive of a CW capability. Evaluating the effectiveness of the verification regime for the Chemical Weapons Convention must take into the account the specificity and sensitivity of these various signatures and how much confidence one might have in them. The following analysis does not attempt a full assessment of the CWC verification regime (e.g., detailed procedures for inspections) but focuses more narrowly on the utility of the various signatures that might be monitored.

A separate but related issue is the quality of the evidence needed to ‘prove’ to the international community that a country has violated its treaty obligations, and the consequences of detecting a violation. This issue of the standard of proof has been a long-standing problem of verification.

Although there may be sufficient evidence to convince some countries that a violation has occurred—particularly if they are suspicious to begin with—the case may not be unassailable in the face of the accused party’s plausible denials. At the same time, the accusing party may not wish to release all of its supporting evidence to a larger audience because of the risk of compromising sensitive sources and methods of intelligence. The standard-of-proof problem has no simple solution and should be kept in mind during the following discussions of “signatures” of chemical weapon acquisition.

### | Research and Development Signatures

The first stage in the acquisition of a CW capability is laboratory research and development of offensive agents, although this step is not necessary if standard agents and known production processes are to be employed. The following step is pilot-scale production to work out problems in the manufacturing process. Because of the small scale of these operations, they can be very difficult to detect.

#### SCIENTIFIC PUBLICATIONS

One way of tracking a country’s research and development activities relevant to CW is to read its contributions to the chemical literature. The fact that leading academic chemists suddenly stop publishing may be an indicator of military censorship or the diversion of civil scientists into defense work. Publication tracking can also produce red herrings, however, since changes in scientific productivity may result from many factors. During World War II, for example, the Germans read great significance into the fact that references to new pesticides suddenly disappeared from U.S. scientific journals. German intelligence analysts deduced correctly that military censorship was responsible for the cut-off, but they wrongly assumed that the United States had independently discovered nerve agents. The Germans’ faulty intelligence assessment led them to fear U.S. retaliation in kind if they initiated the

use of nerve agents, and was one of several factors that deterred them from resorting to chemical warfare.<sup>67</sup>

While publication tracking might provide clues to technologically advanced CW developments, it would be much less useful in the case of a developing country like Iraq that is simply attempting to produce standard agents with known production processes. Such a country would employ mainly industrial chemists and chemical engineers, who publish very little in the open literature. For this reason, publication tracking is likely to be of only secondary value in monitoring CW proliferation.

#### HUMAN INTELLIGENCE (AGENTS OR DEFECTORS)

Human agents, defectors, or even leaks to the press in more open societies can be of value in revealing the existence of secret chemical-warfare R&D activities. In October 1992, for example, Vil Mirzayanov, a Russian military chemist, gave interviews to the press in which he stated that scientists at the State Union Scientific Research Institute of Organic Chemistry and Technology in Moscow had developed a new binary nerve agent that, in terms of its combat characteristics, was “five to eight times superior” to the most toxic of the VX-type agents now in existence. Mirzayanov also alleged that a batch of between five and 10 metric tons of the new agent had been produced.<sup>68</sup> He was subsequently arrested by the Russian Security Ministry (the successor to the KGB) and charged with revealing state secrets.<sup>69</sup> Because human-intelligence reports—particularly those based on hearsay or indirect evidence—may be misleading, however, they typically need to be confirmed with other, more

objective forms of evidence before being used to support final conclusions.

#### | External Production Signatures

Since so much of CW agent production involves dual-use technologies, it is necessary to distinguish clearly between illicit and legitimate production. Unfortunately, there are few, if any, specific, unambiguous external signatures of CW production. A number of potential indicators are discussed below.

#### PATTERNS OF MATERIAL AND EQUIPMENT IMPORTS

Developing countries seeking to acquire a CW capability are nearly always dependent on outside assistance, at least in the initial stages. During the 1980s, numerous companies from Western Europe and Japan sold chemical plants to proliferant countries, which then converted them into CW production facilities. Different suppliers provided the laboratories and production plants, sold chemical precursors, and furnished maintenance equipment. Iraq, for example, was able to purchase 7 turnkey chemical plants and to order thousands of catalogue parts on the international market, along with all the necessary precursor chemicals for the production of CW agents. Similarly, the Libyan CW plant at Rabta was designed by the West German firm Imhausen-Chemie and built by companies from “nearly a dozen nations, East and West,” according to Robert M. Gates, then deputy director of the National Security Council.<sup>70</sup>

Because of the initial reliance of proliferants on outside assistance, suspicious exports and imports of production equipment and chemical

<sup>157</sup> Harris and Paxman, *op. cit.*, footnote 25, p. 64. For a discussion of other factors that convinced the Germans not to use chemical weapons, see Frederick J. Brown, *Chemical Warfare: A Study in Restraints* (Princeton, NJ: Princeton University Press, 1968); and John Ellis van Courtland Moon, “Chemical Weapons and Deterrence: The World War II Experience,” *International Security*, vol. 8, No. 4, spring 1984, pp. 3-35.

<sup>68</sup> “Mirzayanov, Fedorov Detail Russian CW Production,” *Op. Cit.*, footnote 34, p. 2.

<sup>69</sup> Serge Schmemmann, “K.G.B.’s Successor Charges Scientist,” *New York Times*, Nov. 1, 1992, p. 4.

<sup>70</sup> William Tuohy, “U.S. Pressing Allies on Libya Chemical Plant,” *Los Angeles Times*, Jan. 3, 1989, p. 10.



precursors may indicate the acquisition of a CW production capability. For this reason, monitoring exports of materials considered critical for CW production, such as glass-lined pipes and corrosion-resistant alloys, could prove useful.<sup>71</sup> Since much of this equipment is dual-use, however, its acquisition is not necessarily proof of an intent to produce CW agents. Moreover, tracking such transactions is difficult because proliferants like Iraq and Libya take care to set up elaborate networks of ‘front’ companies, paper subsidiaries, and middlemen to hide their purchases.

Precursors of CW agents are also difficult to track. Unlike weapon-grade fissionable materials (e.g., highly enriched uranium and plutonium), which are produced in relatively small quantities and have quite restricted civil uses, most CW precursors have legitimate commercial applications and are traded internationally in volumes that make precise accounting impossible. A useful case study is that of thiodiglycol, the immediate precursor of sulfur mustard. Since a limited number of companies and countries manufacture this chemical, it was initially believed that calculating the agreement between its production and consumption, or *material balance*, might provide a way to detect diversions from legitimate commercial uses to illicit mustard production.

In 1989-91, a working group of the international scientists’ organization Pugwash studied the feasibility of such a monitoring effort. They found that since thiodiglycol could be produced secretly or diverted from legitimate uses with relative ease, an effective control regime would require: 1) continuous monitoring of all chemical plants capable of producing it, and 2) establishing a materials balance between starting materials and products at all stages of its life-cycle. The Pugwash team concluded that such a monitoring system would be extremely difficult and costly to

implement. Moreover, standard inaccuracies in data-gathering on feedstock chemicals could mask the diversion of significant quantities of thiodiglycol for the production of mustard agent, rendering mass-balance calculations of questionable utility.<sup>72</sup>

Tracking phosphorus-based compounds used to make nerve agents is even more difficult. Billions of pounds of these chemicals are bought and sold for commercial purposes, so that militarily significant quantities would be lost in the ‘noise’ of international trade. Because the production of many basic commodity chemicals is shifting from the industrialized countries to the developing world, some precursor chemicals are produced at multiple locations in several countries, greatly complicating the difficulty of material accounting. Moreover, given the long interval between order and delivery, it is difficult to account for materials in transit.

At the level of an individual plant, calculating the material balance between the feedstocks entering a plant and the products coming out is only possible to an accuracy of 2 to 3 percent—a margin of error too large to prevent a militarily significant diversion of precursors to CW agent production. Calculating a precise material balance would also require extensive access to a company’s production records and might therefore jeopardize legitimate trade secrets. As a result, materials-balance calculations cannot provide a reliable indicator that precursor chemicals are being diverted to CW agent production.

Some analysts have suggested that the *ratios* of starting materials and catalysts needed for CW agent production might provide reliable signatures because they are distinctive from those used in commercial production. For example, a plant producing 10 tons of sarin per day would need large quantities of precursors and catalysts in

<sup>71</sup> Robert Gillette, “Verification of Gas Plant a Murky Task,” *Los Angeles Times*, Jan. 5, 1989, p. 5.

<sup>72</sup> Martin M. Kaplan et al., “Summary and Conclusions,” *Verification of Dual-Use Chemicals Under the Chemical Weapons Convention: The Case of Thiodiglycol*, S. J. Lundin, ed. (Oxford, England: SIPRI/Oxford University Press, 1991), pp. 124-136.

specific proportions. Nevertheless, many complex factors influence the quantities of precursor chemicals consumed by a chemical plant, including the stoichiometry of the reactions used for agent production, the number of production steps, and the yield of each step.<sup>73</sup> Some reactants might be used in excess to boost yield or to increase reaction rates, and feedstocks could be deliberately stockpiled to distort the calculated ratios. For these reasons, ratios of starting materials are unlikely to provide a reliable indicator of CW agent production.

The presence of key additives (e.g., stabilizers, thickeners, or freezing-point depressants) in association with precursor chemicals may be indicative of CW agent production. Because the use of additives is generally optional, however, their absence would not necessarily rule out illicit CW agent production.

#### ECONOMIC DISLOCATIONS

The clandestine diversion of a large commercial chemical plant (and associated precursor materials) to CW production might have a noticeable impact on the local economy in a small, underdeveloped country with relatively little economic activity. For example, temporarily ceasing civilian production might create observable shortages of consumer goods normally produced by the plant, such as pesticides or drugs. Whether such economic dislocations would be observable, however, depends on the extent to which the chemical plant was integrated into the local economy. In more industrialized countries such as India, South Korea, and Taiwan, such relatively small economic effects would be obscured by the “noise” of fluctuating output within the overall economy. Moreover, in some developing countries, chemical production is

entirely for export, so that one would have to monitor foreign sales rather than domestic markets. A proliferant country might also stockpile a portion of its output for several months or years and use it to make up for shortfalls in normal production. As a result, economic dislocations are unlikely to be a reliable signature.

#### VISUAL SIGNATURES

Unlike nuclear weapon facilities, which are single-use, limited in number, and easy to identify, civilian chemical plants are two orders of magnitude more numerous, have multiple uses, and are configured in different ways depending on the chemical process. Moreover, there are no unique features or external markings that would distinguish a facility capable of CW agent production from an ordinary chemical plant.<sup>74</sup> A clandestine military production facility might be hidden underground or inside a mountain, or embedded within a legitimate chemical complex, making it essentially invisible from the air. Chemical munitions are small, impossible to distinguish visually from high-explosive shells, and easy to conceal, as are bulk chemical agents. Indeed, 100 tons of nerve agent—a militarily significant quantity in many conflict scenarios—would fit into a dozen trailer-trucks.<sup>75</sup> A proliferant country might therefore hide chemical weapons or bulk agent on railcars, in underground bunkers, or in inconspicuous buildings.

Despite these limitations, however, a pattern of anomalous visual indicators at a chemical facility might arouse suspicions that could be verified by other means. Indicators of CW production that might be visible in overhead imagery obtained by reconnaissance aircraft or satellites include:

<sup>73</sup> Mark F. Mullen, Kenneth E. Apt, and William D. Stanbro, *Criteria for Monitoring a Chemical Arms Treaty: Implications for the Verification Regime* (Los Alamos, NM: Los Alamos National Laboratory, Center for National Security Studies, Report No. 13, December 1991), p. 8.

<sup>74</sup> Briefing by Manuel L. Sanches, System Planning Corp., Oct. 23, 1992.

<sup>75</sup> Kathleen C. Bailey, “Problems With a Chemical Weapon Ban,” *Orbis*, vol. 36, No. 2, spring 1992, p. 244.

- the construction of a large chemical plant that has not been reported in the chemical trade press;<sup>76</sup>
- siting of the plant in an extremely remote and isolated location;
- a high level of security surrounding the plant, such as multiple or electrified fences, air-defense batteries, and guard units;
- an extremely dispersed layout, with large distances between buildings to complicate attacks from the air;
- the proximity of the chemical plant to a metal-machining factory capable of fabricating munitions;
- the presence of tanker trucks associated with the transport of hazardous chemicals;
- a lack of steel drums or other packaging materials normally associated with the production of commercial chemicals;
- traffic movement at night or under guard;
- the death of vegetation, livestock, birds, or wild animals in the vicinity of a plant;
- a flurry of activity at a chemical facility suggestive of a major accident, yet no coverage of the event in the local media.

Box 2-A provides an example, taken from press accounts, of how visual signatures contributed to the identification of a CW production facility in Libya. The utility of these signatures depends, of course, on the individual proliferant's approach. Although the visual indicators cited above would have worked well for Iraq, they would have been unsuccessful in the case of a proliferant that

embedded its CW production facilities within large commercial chemical complexes.

In addition to visual signatures, infrared reflections or emissions detected by specialized overhead sensors might, in principle, provide indications of CW production activity.<sup>77</sup> For example, multispectral cameras might detect stressed or dying foliage around a production plant resulting from emissions of toxic chemicals. A thermal-infrared camera might also assist in monitoring a facility's operational status by revealing the intensity of heat emitted by various parts of the production line and smokestacks. Cool and hot buildings, pipelines, and tanks could be readily identified by infrared imaging, indicating which parts of the facility were active at any given time—although such evidence could be misleading.<sup>78</sup> Synthetic-aperture radar (SAR) imagery, which tends to highlight reflective surfaces, might also reveal details not evident in an optical photograph such as feed pipes, power lines, and vehicles parked around a facility.<sup>79</sup> SAR can also generate images of near-photographic quality at night and in bad weather.

Nevertheless, overhead reconnaissance has its limitations. In addition to the difficulty of distinguishing a CW production facility from an ordinary chemical plant in photographs, overhead imagery may be susceptible to deliberate efforts at camouflage, concealment, and deception (CC&D). The Libyans, for example, reportedly engaged in an effective deception campaign that initially convinced Western intelligence agencies that the Rabta CW production plant had been destroyed

<sup>76</sup> In general, the trade press reports on the construction of all major legitimate chemical plants.

<sup>77</sup> For additional information on surveillance and remote sensing, see U.S. Congress, Office of Technology Assessment, *Cooperative Aerial Surveillance in International Agreements*, OTA-ISC-480 (Washington, DC: U.S. Government Printing Office, July 1991) and U.S. Congress, Office of Technology Assessment, *The Future of Remote Sensing From Space: Civilian Satellite Systems and Applications*, OTA-ISC-558 (Washington, DC: U.S. Government Printing Office, July 1993).

<sup>78</sup> Amy Smithson and Michael Krepon, *Strengthening the Chemical Weapons Convention Through Aerial Inspections*, Occasional Paper No. 4 (Washington, DC: Henry L. Stimson Center, April 1991), p. 15.

<sup>79</sup> Synthetic-aperture radar (SAR) creates a detailed terrain map by capturing the reflection of timed microwave pulses emitted by a moving transmitter, whose motion creates a synthetic antenna with an apparent diameter much greater than that of the actual transmitting antenna. This large effective antenna size allows much greater resolution than a stationary radar could achieve, permitting the creation of photograph-like images.

### Box 2-A-How Libya's Secret CW Plant Was Detected

Press accounts provide an interesting illustration of how patient detective work by the U.S. intelligence community, compiling data from a wide variety of sources, provided strong circumstantial evidence that Libya was building a clandestine CW facility long before the plant started production.<sup>1</sup> (In summarizing these stories here, OTA is neither confirming nor challenging their accuracy.) The Rabta case therefore provides some useful lessons in the detection of covert CW proliferation by intelligence means.

In the early 1930s, reconnaissance-satellite photos of Libya revealed that a major construction project was under way in a hilly region about 35 miles southwest of the Libyan capital of Tripoli. Western intelligence reports also indicated that Ihsan Barbouti, an Iraqi-born businessman whose Frankfurt-based engineering firm had been linked to the construction of a CW plant in Iraq, was using front companies to ship chemical equipment, supplies, construction plans, and personnel to Libya. Barbouti's operation involved some 30 German companies, several Austrian engineers, and Swiss banks.<sup>2</sup>

The prime contractor was Imhausen-Chemie, a West German chemical firm that became involved with the Libyan project in 1985, at a time when it was in financial difficulties. Most of the equipment and supplies left European ports under false export documents, and in order to circumvent existing export controls, Barbouti used a complex commercial network involving front companies that transferred goods through ports in the Far East.<sup>3</sup> Construction at Rabta was carried out under tight security conditions by 1,300 low-wage laborers imported from Thailand.<sup>4</sup>

Meanwhile, satellites and high-altitude reconnaissance aircraft followed the progress of construction at the Rabta site. By 1938, the imagery suggested that the facility, which sprawled over several acres, was nearing completion. Libyan government officials adamantly insisted that the Rabta facility was a pharmaceutical plant, designated Pharma-150. Yet the factory was unusually large by the standards of the pharmaceutical industry and was ringed by high fences and 40-foot sand revetments—seemingly excessive security for an ordinary chemical plant.<sup>5</sup> Since the production facility was completely enclosed inside a warehouse-like structure, overhead photography revealed nothing about the process equipment inside, but the plant's oversized air-filtration system suggested that it was intended for the production of toxic chemicals.

Once the overhead imagery had aroused suspicions, Western countries sought to develop new sources of information among the foreign technicians and construction workers from more than a dozen European, Asian, and Middle East countries employed at the Rabta facility. These sources described plant equipment layout, and supplies, providing additional clues that the site might be intended for CW production. Intelligence analysts concluded that the complex comprised a large chemical agent production plant, a chemical arms storage building, and a metalworking plant built by Japan Steel Works.<sup>6</sup> The latter facility contained Japanese-made machine tools, officially intended for the production of irrigation pumps but also suitable for the production of artillery shells and gas cannisters.<sup>7</sup> Delivery of special steels used in bomb casings suggested to U.S. and British intelligence that

<sup>1</sup> See Thomas C. Wiegale, *The Clandestine Building of Libya's Chemical Weapons Factory: A Study in International Collusion* (Carbondale, IL: Southern Illinois University Press, 1992).

<sup>2</sup> William C. Rempel and Robin Wright, "Libya Plant Found by Vigilance, Luck," *The Los Angeles Times*, Jan. 22, 1989, pp. 1, 21.

<sup>3</sup> Timothy Aepfel, "Seeking Smoking Guns," *The Christian Science Monitor*, Jan. 6, 1989, p. 1.

<sup>4</sup> William R. Doerner, "On Second Thought," *Time*, vol. 133, No. 4, Jan. 23, 1989, p. 31.

<sup>5</sup> Bill Gertz, "Satellites Spot Poison-Bomb Plant in Libya," *Washington Times*, Mar. 5, 1991, p. A3.

<sup>6</sup> Bill Gertz, "12th Chemical Arms Plant Spied in Libya," *The Washington Times*, June 18, 1990, p. A6.

<sup>7</sup> Robert Gillette, "Verification of Gas Plant A Murky Task" *Los Angeles Times*, Jan. 5, 1989, p. 11.

### Libya was actually manufacturing chemical munitions?

The West German government obtained construction blueprints of the Rabta plant from the engineering firm Saltzgitter. These plans revealed some anomalous features suggestive of CW agent production. According to a German government report,

The joint planning of chemical plants and the metal processing plant as well as security facilities not usually found in a pharmaceutical facility (airtight windows and doors, gas-tight walls between the production and the control unit, burn-off unit, corrosion-proof lining on pipes, and escape routes) make it possible to draw the conclusion that 'Pharma 150' is a chemical weapon Plant<sup>8</sup>

It was not until August 1988, however, that the CIA obtained more solid evidence that the Rabta plant was engaged in CW agent production. Following a partial test run of the production process, an accidental spill occurred as highly toxic wastes were being transferred for disposal outside the plant. The resulting cloud of fumes killed **a pack of wild desert dogs** in the vicinity of the plant. Their bodies, detected by satellite, indicated that the plant was producing chemicals of warfare toxicity.<sup>10</sup>

The "smoking gun," however, reportedly came from communications intercepts. During the accident, panicked Libyan officials called Imhausen-Chemie—the West German firm that had designed the plant—for emergency advice. Since the Libyans placed the call over international phone lines, U.S. intelligence was able to intercept the conversation.<sup>11</sup> According to an account in *Time* magazine, "in a frantic effort to get advice on cleaning up and repairing the plant, Libyan officials spoke at length with Imhausen-Chemie personnel. Those conversations left no doubt that employees of the West German firm were just as aware as the Libyans that the plant was being used to produce toxic gas."<sup>12</sup>

On September 14, 1988, the State Department went public with the following statement: "The U.S. now believes Libya has established a CW production capability and is on the verge of full-scale production of these weapons." CIA director William Webster provided further details in a speech *on* October 25, 1988 claiming that the Libyan plant was the largest chemical weapon facility the agency had detected anywhere in the developing world.<sup>13</sup> In August 1990, the intelligence community deduced that large-scale production of chemical agents at Rabta had begun after a photoreconnaissance satellite observed specialized trucks designed to transport CW agents picking up barrels of suspected agent at the plant.<sup>14</sup> In 1992, an intelligence official stated publicly that the Rabta facility had produced and stockpiled more than 100 metric **tons of the nerve gas sarin and other CW agents**.<sup>15</sup>

**Nevertheless, the public case against Rabta—as reported in the news media—is circumstantial and will remain so until Libya signs and ratifies the Chemical Weapons Convention and permits intrusive inspections of the facility.** According to a skeptical assessment, "Neither the charges that Libya is attempting to develop chemical weapons nor the allegations that Libyan forces have used them can be independently substantiated from the public

<sup>8</sup> James Adams, *Engines of War: Merchants of Death and the New Arms Race* (New York, NY: Atlantic Monthly Press, 1990), p. 243.

<sup>9</sup> "Report Submitted by the Government of the Federal Republic of Germany to the German Bundestag on Feb. 15, 1989, Concerning the Possible Involvement of Germans in the Establishment of a Chemical Weapon Facility in Libya," reprinted in U.S. Senate, Committee on Foreign Relations, *Chemical and Biological Weapons: The Urgent Need for Remedies*, 101st Congress, First Session, Jan. 24, Mar. 1, and May 9, 1989 (Washington, DC: Government Printing Office, 1989), p. 81.

<sup>10</sup> Rempel and Wright, *op. dt.*, footnote 2, p. 21.

<sup>11</sup> *Ibid.*, pp. 1, 21.

<sup>12</sup> Doerner, *op. dt.*, footnote 4, p. 31.

<sup>13</sup> David B. Ottaway, "Behind the New Battle With Libya," *The Washington Post*, Jan. 8, 1989, p. C4.

<sup>14</sup> Gertz, *op. cit.*, footnote 6, p. A1.

<sup>15</sup> Gordon C. Oehler, "Address to the Annual Soref Symposium of the Washington Institute for Near East Policy," Apr. 27, 1992, p. 4.

### Box 2-A-How Libya's Secret CW Plant Was Detected-(Contfnued)

record, but there is sufficient circumstantial evidence to make Libya a strong suspect as a chemical weapons proliferator.<sup>16</sup>

Moreover, although the detection and monitoring of the Rabta site was an intelligence success-story, it remains to some extent a special case. Because Libya is a desert nation that relies heavily on **foreign expertise and labor**, the presence of foreigners **provided valuable sources of information**. **Densely populated and industrialized countries** suspected of having covert CW programs are harder to monitor because they can conceal them in a large and diverse array of chemical plants involved in production of pharmaceuticals, pesticides, and fertilizers.<sup>17</sup> Finally, even in the Rabta case, the inherent unreliability of circumstantial evidence underscores the importance of rigorous onsite inspection in verifying the Chemical Weapons Convention.

<sup>16</sup> Gordon M. Burck and Charles C. Flowerree, *International Handbook on Chemical Weapons Proliferation* (New York, NY: Greenwood Press, 1991), p. 267.

<sup>17</sup> Rempel and Wright, op. dt., footnote 2, p. 21.

by fire on March 13, 1990.<sup>80</sup> When the French commercial Earth-resources satellite SPOT-1 photographed the Rabta facility on March 18, however, it looked fully intact.<sup>81</sup> According to press reports, only after several days did the U.S. intelligence community realize that the Libyans had created the illusion of a major fire at the plant by painting scorch marks on the roofs of buildings, burning several truckloads of old tires to produce black smoke, and rushing ambulances to the area to make it appear that the plant had suffered severe damage.<sup>82</sup>

In sum, external visual signatures, such as those that might be observed through overhead photography, can provide clues of CW production activities but are rarely conclusive and must be supplemented with evidence from onsite inspections. While it is possible to conclude from indirect or ambiguous signatures that something suspicious is going on, making a convincing case that a country has broken its

solemn treaty commitments requires a higher standard of evidence.

#### | Internal Production Signatures

Under the CWC verification regime, external signatures obtained noncooperatively through overhead photography and remote-sensing will be supplemented with internal signatures obtained by authorized onsite inspections. Examples of some internal signatures are discussed below.

#### PRODUCTION PROCESS EQUIPMENT

As discussed above, the synthesis of nerve agents requires a few reactions that are rare in the production of pesticides: the cyanation reaction for the synthesis of tabun; the alkylation reaction for the synthesis of sarin, soman, and VX; and the fluorination reaction for the synthesis of sarin and soman.<sup>83</sup> Indeed, since alkylation is not required for the production of most organophosphorus pesticides, civil plants employ feedstocks con-

So Michael R. Gordon, "U.S. Says Fire at Libya Arms Plant May Be a Hoax," *The New York Times*, Mar. 31, 1990, p. 3.

<sup>81</sup> "Small Fire, Much Smoke," *The Economist*, vol. 314, No. 648, Mar. 31, 1990, p. 42.

<sup>82</sup> Bill Gertz, "Satellites Spot Poison-Bomb Plant in Libya," *The Washington Times*, Mar. 5, 1991, p. 3.

<sup>83</sup> Burck, op. cit., footnote 33, p. 155.

<sup>84</sup> The phosphorus used in most pesticides is pentavalent rather\* trivalent.

taining a different chemical form of phosphorus than is used to make nerve agents.<sup>84</sup>

Unfortunately, there is no “signature equipment for the manufacture of CW agents. Since CW agents can be and have been produced by a variety of standard organic-chemical processes, it is almost impossible to identify an individual piece of equipment that has been specifically designed or modified for this purpose. This fact makes it extremely difficult for all but the most trained eye to spot a CW production facility. The necessary equipment would tend to be standard rather than unique, consisting of chemical reaction vessels and “back-end” processing equipment. Distillation columns, for example, are not necessarily a good indicator of illicit activity because they are also found in many legitimate chemical plants. They might also be omitted from a CW production facility if the proliferant does not require pure agent with a long shelf-life.

Still, a combination of subtle changes in plant design and layout might be indicative of illicit production, particularly if an analysis of the design suggests that it does not make engineering and economic sense for its declared commercial purpose. For example, unusual process steps such as alkylation might stand out if they are inconsistent with the plant’s present mix of commercial products or are not being carried out on an appropriate scale. A plant designed to work with highly toxic materials might also have specialized pumps and valves with double seals and other safety measures. In such cases, a more intrusive inspection would be warranted to verify that the suspect facility is not engaged in CW production activities.

Another feature of a nerve-agent production plant that might help distinguish it from an ordinary pesticide plant is the means of heating and cooling the reaction vessels. Since chemical processes for nerve agents produce highly unsta-

ble intermediates that react explosively with water, steam-heating and water-cooling must be replaced with special heat-exchange fluids and heating oils that require the use of cooling towers rather than steam vents. A nerve-agent production plant would therefore lack the steam clouds that are a common feature of chemical plants.<sup>85</sup> Even so, this signature would not necessarily be unique to nerve-agent production, since many legitimate chemical plants use organic solvents or mineral oils as heating and cooling media rather than steam or water. Moreover, a shrewd proliferant seeking to avoid detection might deliberately install misleading steam-cloud generators!

In general, analysis of plant design and layout is most useful in the case of turnkey plants developed and exported by foreign companies, which tend to use distinctive design formats and templates. Indigenously designed chemical plants may have unique layouts that make it more difficult to draw inferences about their functions.

#### CORROSION-RESISTANT MATERIALS

Since the reactions needed to produce mustard and nerve agents are highly corrosive, long-term CW production facility might use corrosion-resistant pipes, valves, and reaction vessels made of special alloys with a high nickel content, such as Hastelloy. Unfortunately, there is currently no practical method to identify corrosion-resistant materials without taking physical samples or looking inside, particularly if a reactor or pipe is painted or wrapped in insulating material.<sup>86</sup> Moreover, the use of corrosion-resistant reactors and pipes is increasingly common in the civilian chemical industry. Since commercial manufacturers may wish to avoid replacing vessels and pipes on a regular basis, some advanced commercial plants build in an extra level of protection by installing Hastelloy or glass-lined reaction vessels to protect equipment and maintain product

<sup>85</sup>Stockholm International Peace Research Institute, *The Problem of Chemical and Biological Warfare*, Vol. VI: *Technical Aspects of Early Warning and Verification* (Stockholm: Almqvist & Wiksell, 1975), p. 293.

<sup>86</sup>Sanches et al., op. Cit., footnote 66, p. 63.

purity. A proliferant country might also be able to acquire used corrosion-resistant equipment that still has a few years of life in it after the guaranteed 5 to 10 years have expired.<sup>87</sup>

Conversely, a proliferant engaged in the covert production of CW agents might choose deliberately not to use corrosion-resistant materials for the following reasons:

- such materials might not be available because of export controls;
- the use of such materials might reveal the intent to produce CW agents; or
- the near-term capital cost to extend the life of the equipment would not be justified, particularly if a country planned to produce only a limited stockpile of chemical weapons.

Although a stainless-steel reactor will be severely corroded by HF gas, it can still function for about a year. A proliferant might therefore be willing to live with the inconvenience of replacing equipment at shorter intervals and use ordinary construction materials in an attempt to conceal its activities. For this reason, the presence in a chemical plant of corrosion-resistant material does not necessarily indicate that CW agents are being produced, and its absence from a suspect facility may merely reflect the frequent replacement of standard equipment or a lack of plans for long-term agent production.

#### SAFETY AND POLLUTION-CONTROL EQUIPMENT

The toxicity of nerve agents is roughly 1,000 times greater than that of most organophosphorus pesticides. Nevertheless, only the last step in agent production poses a serious toxic hazard; in the case of both G and V agents, this is a very small part of the process. To prevent the release of deadly fumes into the environment, the final process step in a CW production plant would

probably be carried out in a tightly sealed enclosure, operated at a negative pressure so that any leaks would result in air being drawn in rather than toxic gases escaping. Reaction vessels involved in this step might also be operated by remote control, requiring special piping and computer systems, and pumps might be equipped with double or triple seals to guard against leaks.<sup>88</sup>

A CW production plant might also have ventilation and emission-control systems that differ from those of a legitimate pesticide or pharmaceutical plant. In pesticide plants, fresh air often circulates continually through the plant and vents directly into the atmosphere. Although increasingly stringent environmental regulations are strengthening emission controls in developed countries, pesticide plants in developing countries are likely to be open to the environment.<sup>89</sup> Similarly, pharmaceutical plants generally shield products from contamination by maintaining the production area at a *higher air* pressure than the outside environment, so that all contaminants flow away from the production process. In contrast, in a CW agent plant the final production steps would probably be maintained at a *lower* pressure than the outside air so that the lethal vapors do not leak into the surrounding environment.

The hazards associated with production of nerve agents might also require the use of large activated-carbon filtration systems and scrubbers to remove all supertoxic chemicals from the exhaust air. The German firm of Noske-Haeser, for example, installed an expensive air-cleaning plant for the Iraqi chemical laboratory at Salman Pak. Intelligence analysts concluded that the dimensions of the air-cleaning system were too large if the 10 laboratories were simply engaged in commercial research and development, partic-

<sup>87</sup> Gordon Burck, EAI corporation personal communication, 1992.

<sup>88</sup> Burck, op. cit., footnote 13, p. 129.

<sup>89</sup> Peter M. Zapf, "Appendix A: The Chemistry of Organophosphate Nerve Agents," Benoit Morel and Kyle Olson, eds., *Shadows and Substance: The Chemical Weapons Convention* (Boulder, CO: Westview Press, 1993), p. 297.



ularly given the fact that the Iraqis did not normally care about environmental protection.<sup>90</sup> Thus, while plant emission controls would reduce chemical signatures outside the plant, the presence of scrubbers and other air-cleaning systems would provide a clue that toxic agents were being produced.

Still, while special containment measures may provide a telltale sign of CW agent production, they are by no means a foolproof signature. First, the pressurization of a facility can be reversed by changing the direction of air flow, perhaps in a deliberate attempt to deceive an inspection team, although this capability must be designed-in. Second, as the chemical industry has adapted to increasingly stringent environmental and occupational-safety laws, ordinary chemical plants have increasingly adopted sophisticated air-treatment systems and corrosion-resistant materials, blurring the distinction between CW-capable and commercial facilities. As a result, equipment designed for commercial purposes may provide adequate containment for CW-agent production.<sup>91</sup>

Conversely, a lack of stringent safety measures is not a foolproof indicator that a country is not producing CW agents, since a ruthless government that does not care about the welfare of workers might fail to take such basic precautions. Although the German Government argued that the chemical plant sold to Iraq by the Karl Kolb firm was not suitable for CW production because it lacked adequate safety equipment, the real reason for the lack of safety measures was Saddam Hussein's willingness to tolerate a high incidence of injuries and deaths among the plant staff. Iraqi officials later admitted to UN inspectors that there were about 100 accidents per year involving chemical agents, 10 of them major.<sup>92</sup>

Finally, the advent of binary chemical weapons means that it is no longer necessary to manufacture supertoxic agents to acquire a CW capability. Instead, a production plant could manufacture DF, the immediate precursor of the G agents, which is no more toxic than many commercial organophosphorus pesticides. Such a plant would not require high levels of containment and hence could be more easily disguised.

#### WASTE TREATMENT AND DISPOSAL

Chemical weapon producers have been known to dispose of their highly toxic wastes in an environmentally reckless manner. After World War II, the Soviet Union and other countries dumped large quantities of nerve and mustard agents at sea in metal barrels that have now corroded, posing a serious threat to the marine environment. The Soviets also dumped vast quantities of toxic wastes from CW agent production directly into rivers. In the aftermath of the Gulf War, Iraq destroyed large quantities of chemical munitions it had failed to declare by pouring the toxic agents into standing ponds or holes dug in the ground, and by open-air burning.<sup>93</sup>

Nevertheless, countries with greater concern about protecting the environment might equip a CW agent production plant with more extensive waste-treatment facilities than a typical commercial plant. Such facilities might include tanks for the storage of toxic wastes and a treatment unit to neutralize acid byproducts with alkaline chemicals and to detoxify and remove phosphorus compounds. After treatment, the neutralized wastes might be reduced by evaporation or incineration, and then disposed of in ways that might be observable. For example, *waste lagoons* are quite conspicuous because of their size and

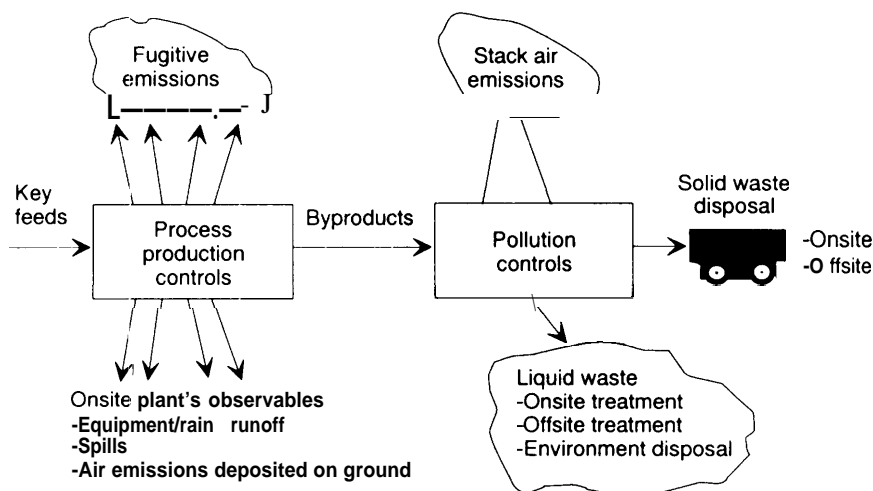
<sup>90</sup> "Report on German Technology in 'Diyala' Gas Lab," *Stern*, Feb. 7, 1991, pp. 29-33; translated in FBIS-WEU-91-032-A, Feb. 15, 1991, p. 9.

<sup>91</sup> Burck and Flowerree, op. Cit., footnote 14, p. 13.

<sup>92</sup> *Chemical Weapons Convention Bulletin*, op. cit., footnote 50, p. 16.

<sup>93</sup> Peter Grier, "UN Inspectors in Iraq Get Chemical Surprise," *Christian Science Monitor*, June 23, 1992, pp. 1,4.

Figure 2-4-Production Facility Observable



SOURCE: System Planning Corp., *Chemical Weapons Convention (CWC) Signatures Analysis*, Final Technical Report No. 1396, August 1991, p. 61.

because phosphates promote algal and bacterial growth, which would be visible in overhead photographs. (However, some legitimate chemical plants have lagoons situated near agricultural land, where fertilizer runoff may cause similar algal blooms during the summer months.) Another disposal method involves injection of toxic wastes into *deep underground wells*. Since deep wells are hard to dig and would have to be quite large, they might be difficult to conceal.<sup>94</sup> The waste well at the U.S. Rocky Mountain Arsenal, for example, was 12,000 feet below the surface.

### CHEMICAL SIGNATURES

The goal in collecting and analyzing samples during on-site inspections of chemical plants and suspect facilities is to detect signatures of illicit CW production; at the same time, it is important to minimize the potential for false alarms and to limit the disruptive effects of sampling on the commercial chemical industry. Phosphorus-methyl (P-CH<sub>3</sub>) bonds are characteristic of nerve agents, are rare in most organophosphorus pesti-

cides, and are extremely resistant to degradation and hence persist for long periods in the environment. The phosphorus-fluorine bond found in sarin and soman is also unusual, and its detection in a commercial pesticide plant would warrant further investigation.

Chemical signatures may be detected from a variety of sources. (See figure 2-4.) Inspectors given on-site access to chemical facilities under the terms of the chemical Weapons Convention will be allowed to take wipe samples from the surfaces of process and pollution-control equipment, as well as liquid samples from the production process and the waste stream. Even if the reactors and pipes are flushed clean prior to an inspection, the production of CW agents would leave behind traces of agents, precursors, and byproducts that are absorbed into rubber seals and gaskets, which are too costly to replace frequently. The concrete floor of a plant also provides an absorbent matrix for leaked chemicals and is a potential reservoir of CW agent residues. Analyzing such samples with sensitive

<sup>94</sup> Stockholm International Peace Research Institute, *op. cit.*, footnote 85, p. 293

analytical techniques such as combined gas chromatography/mass spectrometry should therefore reveal the presence of telltale chemicals. (See app. 2-A.) Nevertheless, such sampling and analysis may be constrained by the amount of access provided during an onsite inspection.

### | Detecting Clandestine Production

Detection of clandestine CW agent production in a nondeclared facility would require noncooperative data collection by human agents or by covertly emplaced or remote sensors, which might then be used to cue a challenge inspection. While detection of a clandestine production facility would be difficult, some possible signatures are discussed below.

#### EFFLUENT ANALYSIS

A number of approaches rely on the monitoring of plant effluents to detect clandestine CW agent production at a distance. One such approach is to use computer atmospheric models to predict where gaseous plant emissions are most likely to be deposited on the ground, and to take soil samples from such locations for analysis. Because the atmospheric models are imperfect, however, the effluent sample may be too dilute to be identified.

Near-site monitoring techniques, such as laser spectroscopy, are also under development for detecting telltale chemicals in the exhaust plumes rising from chemical plant stacks without the need to obtain access to a plant site. Such technologies include both passive spectroscopic systems that detect and analyze radiant emissions at multiple wavelengths, and active systems that transmit laser radiation at selected wavelengths and then analyze the backscattered or emitted radiation. (See app. 2-A.) At present, near-site monitoring technology is not yet sufficiently sensitive or reliable for verification purposes. Part of the problem is that the quantity of gaseous emissions from a chemical production facility is very site-dependent and is a function

of the plant's emission-control systems and the quality of its maintenance.

A third approach to detecting clandestine CW agent production is the analysis of liquid effluents. Since no chemical reaction is 100 percent complete, there are always some residual materials left over that may not be emitted as a gas and will emerge in the waste stream. All methods of CW production produce significant quantities of wastes, although the exact amounts depend on the choice of production process, the extent of recycling, and how rapidly the waste stream is sent to a treatment facility. Flushing out the production line with a decontaminating solvent or water also creates a liquid effluent that must be disposed of. Analyzing such chemical traces may therefore provide a means of detecting CW agent production without gaining access to the interior of a site. Nevertheless, effluent analysis has a number of limitations:

- A proliferant may simply store production wastes onsite or inject them into a deep well rather than releasing them into the environment. A proliferant might also create a phoney waste stream to mislead monitors, who would not be able to detect the real fate of the production wastes without access to the interior of the plant.
- Once the waste stream has passed through a treatment facility, its characteristic chemical components may be destroyed. Since a handful of commercial products (pesticides and fire retardants) break down to methylphosphonate, the same final degradation product as nerve agents, merely identifying this compound in the waste stream would not in itself provide conclusive evidence of a violation.
- If the plant effluent were discharged into a river, one would have to obtain water samples close to the source before the chemical signatures were diluted to undetectable levels.

For a detailed discussion of effluent analysis, see appendix 2-A.

### BIOMARKERS IN PLANT WORKERS AND WILDLIFE

Yet another approach to monitoring makes use of the natural ecosystem around a chemical plant as a long-term collection mechanism. In recent years, occupational health specialists have identified a number of “biomarkers” associated with exposure to toxic chemicals such as pesticides. Living plants and organisms (including humans) tend to concentrate various trace chemicals in their tissues, so that measurable quantities can be detected in the higher members of the food chain living in the vicinity of a suspect facility. Entire small organisms (e.g., insects), samples of animal fur, urine, blood, or feces, or plant leaves, flowers, fruit, or roots, could be analyzed to identify chemical compounds not normally present in the local environment.<sup>95</sup> Such an approach might provide more comprehensive coverage than point detectors.

A related approach is to collect samples of tie, blood, skin, or hair from chemical plant workers and analyze them for telltale biomarkers of covert CW production. One might look either for metabolites of sulfur mustard and nerve agents in body fluids such as blood and urine, or for ‘adducts’ of mustard or nerve agents bound to cellular DNA. (See app. 2-A.) In the United States, however, such monitoring might be considered a violation of Fourth Amendment protections against intrusive personal searches if it were conducted without a warrant, and other countries might simply refuse to allow it.

### | Storage of Agents and Munitions

Although CW munitions are indistinguishable at a distance from conventional munitions, they may be stored in bunkers that have distinctive characteristics. In Iraq, CW storage bunkers were located inside ammunition-storage depots but were secured separately with fencing or barbed wire, set off in remote locations, and spaced far

apart. Before the start of the Coalition bombing campaign during Operation Desert Storm, however, the Iraqis moved many of their chemical munitions out of the storage bunkers and buried them in the desert to protect them from attack. The Iraqis also constructed decoy bunkers intended to mislead enemy bombers. Thus, storage bunkers may not be a reliable signature of either the presence or the absence of a CW capability.

Of course, the discovery of stockpiled chemical munitions would provide a clear indication of a CW capability. Artillery shells and rockets containing CW agents, high explosives, or smoke rounds are identical in shape, however, and differ only by an external color code that could be easily painted over. Since chemical munitions are impossible to distinguish by visual inspection alone, a proliferant country might attempt to violate the Chemical Weapons Convention by painting chemical munitions to look like high-explosive shells and storing them in the same depot.

In order to characterize the contents of sealed munitions while avoiding the hazards of direct sampling, several nondestructive evaluation (NDE) methods are under development. One such method, known as Portable Isotope Neutron Spectroscopy (PINS), was developed by the Idaho National Engineering Laboratory. This technique involves irradiating a shell with neutrons, which interact with the chemical contents of the munition to produce gamma rays that are unique for each chemical element. Nerve agents are rich in phosphorus and mustard agents in chlorine, while high explosives contain large quantities of nitrogen but no phosphorus, chlorine, or arsenic. As a result, the gamma emission spectra for CW agents and high explosives are easily distinguishable. Although the PINS technique has shown a 95 percent accuracy rate in tests on known chemical shells, its reliability under uncontrolled field conditions has not yet

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<sup>95</sup> Sylvia Talmage and Barbara Walton, “SXI@ Mammals as Environmental Monitors,” *Oak Ridge National Laboratory Review*, vol. 25, No. 1 (1992), pp. 55-57.

been demonstrated conclusively.<sup>96</sup> Several other NDE systems are also under development and have differing strengths and weaknesses.<sup>97</sup> For example, acoustic resonance spectroscopy uses sound waves to assay the contents of a shell. It has the advantage of being able to complete an assay in 10 seconds, but the disadvantage of requiring the acoustic signatures of known reference shells of the same type.

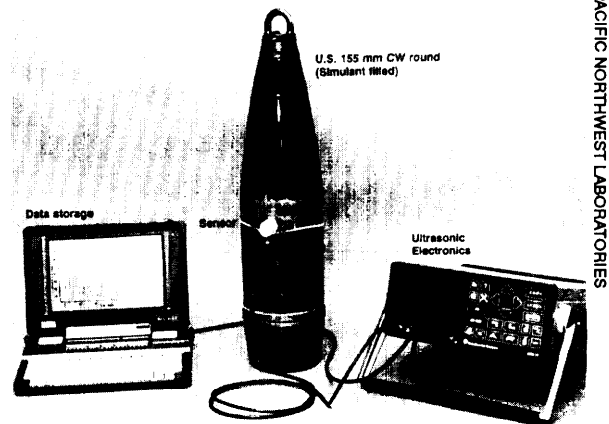
### | Weaponization and Testing Signatures

Weaponization and testing of CW munitions may also provide signatures, although these, too, may be ambiguous.

#### VISUAL SIGNATURES

Test ranges for operational testing of chemical munitions and dual-use delivery systems such as artillery and missiles cannot be hidden underground or inside closed buildings, and hence may show up in overhead images. Such a test range generally consists of a support area containing administration and logistics and an experimental area containing a test grid and a large downwind sampling zone with an array of sampling poles.<sup>98</sup> Nevertheless, an illegal test facility might well be camouflaged and the tests conducted at night or when reconnaissance satellites are out of range. Observing a test might require considerable luck.

Ground or aerial observations of military exercises involving chemical weapons might provide some clues to a country's intentions, but they are probably not a reliable signature. One problem is that it is very difficult to understand the purpose of a military exercise without knowing the scenario that the planners are running. Exercises that involve the firing of munitions to generate an aerosol might imply preparations for



PACIFIC NORTHWEST LABORATORIES

*Ultrasonic pulse echo apparatus to distinguish chemical munitions from conventional rounds. No echo is observed if the shell is empty; echoes will return at characteristic times if the shell contains a solid, liquid, or powder.*

offensive CW use, but they could also pertain to the generation of smoke screens. Alternatively, a proliferant might conduct misleading field exercises for purposes of deception. Defensive and decontamination exercises might also be part of a broader offensive strategy. And although field testing is desirable, it is not an essential prerequisite for acquiring a CW capability.

#### DIFFICULTIES OF DETECTION

The various signatures associated with the acquisition of a CW capability, along with potential detection methods and countermeasures, are summarized in table 2-2. Overall, the challenge of detecting and monitoring clandestine production of CW agents is a formidable one. None of the production signatures is a reliable indicator by itself, and even combinations of signatures may depend on making

<sup>96</sup> Personal communication, A. J. Caffrey, senior scientist, Idaho National Engineering Laboratory, Aug. 23, 1993.

<sup>97</sup> In addition to PINS, other Non-Destructive Evaluation (NDE) methods currently under investigation include x-ray, acoustic resonance spectroscopy, ultrasonic pulse echo, laser acoustic spectroscopy, and ion-tube neutron spectroscopy. For a review of current NDE research and development, see the special issue of *Verification Technologies* devoted to this topic (U.S. Department of Energy, Office of Arms Control and Nonproliferation, *Verification Technologies*, First/Second Quarters 1992).

<sup>98</sup> Sanches et al., op. cit., footnote 66, pp. 53-54.

Table 2-2—Chemical Weapon Program Signatures and Concealment

Program stage	Signature	Detection methods	Concealment methods comment
Design and engineering	Scientific and technical publications (presence or absence)	Literature survey and analysis	<ol style="list-style-type: none"> <li>1. Manage publication activities</li> <li>2. Use widely available technical information rather than design new agents or techniques</li> </ol>
Acquisition of raw materials	Patterns of feed material acquisition	Monitoring of open-source trade data; espionage	<ol style="list-style-type: none"> <li>1. Shuffle, divert acquisitions; mix with legitimate uses</li> <li>2. Develop clandestine networks</li> <li>3. Produce known precursor chemicals indigenously</li> <li>4. With chemical industry development, raw materials acquisitions increasingly lose their utility as a signature</li> </ol>
Clandestine production plant	Security Measures	Overhead imaging or human intelligence (humint)	Conceal measures, or place plant within other secure facilities
	Effluents	Sampling of air, water, or soil near suspect plant—various forms of chemical analysis	<ol style="list-style-type: none"> <li>1. Chemically alter effluents with decontaminating solvents or them with additives</li> <li>2. Hide wastes or remove for off-site disposal</li> </ol>
Converted or multipurpose production plant	Security Measures	Overhead imaging or humint	Conceal measures
	Effluents	Sampling of air, water, or soil near suspect plants—various forms of chemical analysis; laser remote sensing of emission plumes	<ol style="list-style-type: none"> <li>1. Chemically alter or mask effluents</li> <li>2. Remove wastes for offsite disposal</li> </ol>

observations in the right place and at the right time. Major hurdles to detection include:

- the possibility of intermittent production in a small, pilot-scale facility;
- the low volatility of most of the compounds of interest (resulting in low atmospheric concentrations even insignificant leaks occur);
- masking and interference from legitimate chemicals produced at a typical multiple-use facility;
- the political and economic costs of challenge inspections, which will severely constrain the number of facilities that can be inspected;
- the difficulty of detecting production of binary agents, which are made from dual-use

chemicals and widely available industrial alcohols.

Despite the difficulties of detecting clandestine CW production, however, the cooperative verification regime will be supplemented with national intelligence-gathering efforts that may provide indications of CW-related activities somewhere along the acquisition spectrum ranging from research through testing and the development of military doctrine. (As an illustration of the contribution of national intelligence efforts, Box 2-A recounts press reports describing how the United States tracked the Libyan CW production facility at Rabta.) These additional sources of information should increase the chances of detecting a clandestine CW program, and could

Program stage	Signature	Detection methods	Concealment methods comment
	Special safety and containment measures	Onsite inspection of suspect plants	1. Sacrifice worker safety 2. Modern chemical plants increasingly have these features
	Rare chemical processes (e.g., alkylation or cyanation)	Onsite inspection of suspect plants	Alternate weapons agent production with commercial production requiring same processes
	Corrosion-resistant reactors and other fittings	Onsite inspection of suspect plants; tracking of imports of such parts	1. Replace corrodible equipment as needed 2. Trend is toward use of such parts in legitimate commercial processes
	Tell-tale residues within plant	On-site chemical analysis of absorbent parts (or removal for off-site analysis)	1. Use decontaminating solvents 2. Practice quick replacement of such parts as rubber flanges and seals that might absorb residues
	Biomarkers in plant workers	Analysis of urine and blood samples	Prevent collection of samples unless specifically permitted by challenge inspection regime
Weapon assembly	Uniquely configured arsenals (e.g., distribution of storage bunkers)	Overhead imaging	Pattern facilities after conventional arsenals
Weapon testing	Uniquely configured test facilities	Overhead imaging	1. Make special features temporary 2. Test on overcast days, at night, or in absence of imaging devices

SOURCE: Office of Technology Assessment, 1993.

be used to trigger challenge inspections under the treaty regime.

## ALTERNATIVE PROLIFERATION PATHWAYS

There are two basic approaches to acquiring an indigenous CW production capability:

- build a dedicated CW agent production plant (open or clandestine);
- convert existing chemical facilities (single-purpose or multipurpose) to CW agent production on a temporary or permanent basis.<sup>99</sup>

In the past, proliferant countries seeking a CW production capability have purchased turnkey plants from foreign suppliers. For example, both Libya and Iraq purchased entire chemical plants from German firms that were then converted to CW agent production. Increasingly, however, proliferants purchase parts and engineering know-how from a variety of sources and integrate them on their own. This new approach to acquisition makes it more difficult to halt CW proliferation through export controls.

Proliferant countries-particularly those that sign and ratify the Chemical Weapons Convention-

~ A proliferant might also purchase bulk or weaponized CW agents from a state that already possesses them. This approach is likely to be at most temporary, however, since the purchasing state would remain dangerously dependent on the supplier.

are most likely to produce CW agents in a clandestine manner to avoid provoking international political and economic sanctions. Nevertheless, a country that has been threatened or attacked by a more powerful neighbor may seek to acquire a CW capability as quickly as possible. This scenario would be compatible with the manufacture of cheap, low-stability agents for near-term military use and might involve acquiring the capacity for rapid but not necessarily secret production in wartime. Such a ‘breakout’ capability might either be built deliberately in peacetime or improved in response to a military crisis.

### | Building a Dedicated Plant

The advantage of building a clandestine CW plant on a new site is that it can be built in an isolated location, far from commercial chemical plants that might be subject to routine inspections under the Chemical Weapons Convention.<sup>100</sup> The number of plant personnel could be kept to a minimum for security reasons, and specialized construction and camouflage procedures could be used. On the other hand, siting a dedicated CW production facility in the midst of a large commercial industrial complex would have the advantage that the surrounding “noise” would drown out any telltale CW-related signatures. Moreover, the construction of a clandestine plant at an isolated site, if detected, would tend to draw attention to the facility.

Another strategy for a proliferant country would be to acquire one or more pilot-scale

chemical plants and use them to accumulate, over a period of years, enough CW agent to be a potent strategic asset in certain regional conflicts.<sup>101</sup> Because of their small size, pilot-scale facilities would be easier to conceal. Nevertheless, stocks of agent produced over a long period of time would have to be of greater purity to ensure an adequate shelf-life. This requirement would in turn demand distillation and the use of stabilizers, complicating the production process.

### | Converting an Existing Plant

An alternative pathway to acquiring a CW capability would be to convert all or part of a declared commercial facility to CW agent production. Experts disagree over the speed with which a commercial plant could be converted. Former CIA director William Webster alleged that the Libyan Pharm-150 plant at Rabta was capable of CW agent production but that “within fewer than 24 hours, it would be relatively easy for the Libyans to make the site appear to be a pharmaceutical facility.”<sup>102</sup> Reportedly, this potential for deception was one reason that the United States turned down an offer by Libyan leader Muhamar Khadafy to do a one-time onsite inspection of the plant.<sup>103</sup>

Some analysts challenge the assumption that it would be easy to convert a commercial chemical plant to the production of CW agents by simply changing valves or piping.<sup>104</sup> First, only a few types of chemical plants are suitable for conversion to production of nerve agents. Fertilizer plants use a different kind of phosphorus (phos-

<sup>100</sup> Most **commercial** chemical **plants** in the developing world are located in populated areas. Even **if** a commercial plant is **initially built in** a remote **location**, the resulting employment opportunities and economic activity tend to attract large numbers of migrants to the immediate vicinity.

<sup>101</sup> **Robert C. Gough**, Sandia National Laboratory, **personal** Communication 1992.

<sup>102</sup> **Testimony by William H. Webster**, Director of Central Intelligence, in U.S. Senate, Committee on Governmental Affairs, *Global Spread of Chemical and Biological Weapons*, 101st Congress, 1st Session, Feb. 9, 1989 (Washington DC: U.S. Government Printing Office, 1990), p. 13.

<sup>103</sup> **Tuohy**, op. cit., footnote 70, p. 10.

<sup>104</sup> **Much of the following analysis is based** on research conducted by Alan R. **Pittaway** and the Midwest Research Institute **in the late 1960s** and early 1970s. See Midwest Research Institute, *The Difficulty of Converting Pesticide Plants to CW Nerve Agent Manufacture*, Technical Report No. 7 (Kansas City, MO: Midwest Research Institute, Feb. 20, 1970).



phate) chemistry, do not contain most of the necessary equipment for the chemical synthesis of nerve agents, and lack stringent safety and containment measures. Pharmaceutical plants share many precursors with CW agents, but the scale of drug production is generally much smaller than that of other specialty chemicals. Organophosphorus pesticide plants are most suitable for conversion to nerve-agent production, since the phosphorus chemistry is similar and much of the process equipment is of the type and capacity suitable for large-scale production of nerve agents.

In 1987, for example, the United States produced at least 5,000 pounds of each of 204 pesticides, of which 33 were organophosphorus compounds. Of the 33, six were alkylated, making them structurally similar to nerve agents and hence of greatest concern. Those six alkylated pesticides were produced at 24 plants owned by 17 companies.<sup>105</sup> In recent years, however, the trend in pesticide development—at least by countries not seeking to produce nerve agents—has been to move away from alkylated compounds to those with reduced mammalian toxicity. As a result, ever fewer pesticide plants today are equipped with processes that can be readily converted to nerve-agent production, although a proliferant could opt deliberately **for an old production method.**

In addition to pesticides, a handful of commercial organophosphorus compounds are structurally related to nerve agents, including flame retardants, plastics, and fuel additives. Volume of production for the most significant of these compounds, the fire retardant dimethyl methylphosphonate (DMMP), is on the order of 2,200 metric tons annually among four producers world-

wide.<sup>106</sup> Thus, **the manufacture of DMMP and related compounds could still be used as a cover for nerve-agent production.**

The technical hurdles involved in converting a commercial plant to CW agent production are different depending on whether the commercial facility is single-purpose or multipurpose. Both pathways are discussed below.

#### SINGLE-PURPOSE PLANT

Single-purpose chemical plants are generally custom-designed and optimized for production of one product in vast quantities. As a result, converting such a plant to some other form of production can take months. The German pharmaceutical firm Bayer, for example, spent 2 years rebuilding a single-purpose facility so that it could produce two different but related chemicals. After this initial investment, the plant could alternate between the two products with a change-over time of 3 to 4 weeks.<sup>107</sup>

The differences in the chemical synthesis of commercial organophosphorus compounds and nerve agents mean that some of the processes and equipment are not easily convertible, but others are. For example, pesticide plants do not normally contain equipment for performing the cyanation reaction needed for tabun; the alkylation reaction needed for sarin, soman, and VX; or the fluorination reaction needed for sarin and soman. Thus, the presence of any of these process steps in a pesticide plant would warrant further investigation. Pesticide plants normally have distillation equipment that consists mainly of stripping columns, which are not adequate for distilling nerve agents like sarin or soman.<sup>108</sup> (Distillation is only needed, however, if a long

<sup>105</sup> The six pesticides with a P-alkyl bond that were produced in the past in the United States were: 2-chloroethylphosphonic acid, Fonophos, Fosmine ammonium, Glyphosate and its isopropylamine salt, and Trichlorfon. Today, Fonophos is the only alkylated pesticide still produced in significant quantities. See Stanford Research Institute International, *Directory of Chemical Producers USA* (Menlo Park, CA: SRI International, 1988).

<sup>106</sup> Zapf, op. cit., footnote 89, p. 280.

<sup>107</sup> Burck, op. cit., footnote 33, p. 151.

<sup>108</sup> Ibid., p. 148.

shelf-life is required.) Moreover, pesticide plants do not normally use hydrogen fluoride—a key ingredient of sarin and soman—and generally use phosphorus oxychloride ( $\text{POCl}_3$ ) or phosphorus pentasulfide ( $\text{P}_2\text{S}_5$ ) as a starting material rather than phosphorus trichloride ( $\text{PCl}_3$ ). Since phosphorus pentasulfide is not suitable for use in alkylation reactions, it cannot be utilized as a starting material for nerve-agent production.<sup>109</sup>

For these reasons, converting a pesticide plant to nerve-gas production would mean modifying the production process and stretching the operating conditions to obtain reasonable yields while still maintaining secrecy. According to one assessment, for example, “the conversion of a parathion plant to the production of G-agents would be extremely difficult, requiring substantial material changes and plant retooling.”<sup>110</sup> The modifications would involve rerouting pipes, valves, and mechanical seals to meet minimal operating requirements. For example, a proliferant might design a plant to produce an organophosphorus pesticide that lacks a phosphorus-carbon bond and then change the feed materials and process equipment to add a final alkylation step—either in a clandestine section of the main plant or at a separate location. It would also be possible to design a plant that could make nerve agents and then add on “bypass piping to permit the commercial production of pesticides and pharmaceuticals. Thus, in time of need, it would be easy to convert the plant back to nerve-agent production.

Conversion to nerve-agent production might also require upgrading safety, containment, and waste-disposal procedures, although as has been stated earlier, such signatures can be ambiguous. Converting a single-purpose pesticide plant to nerve-agent production would require at least

several weeks and would involve the following steps: design of the modified production line, acquisition of the needed equipment, and construction, checkout, and pilot operation. The actual time requirements would depend on the experience of the plant personnel, the priority given the project, and willingness to cut corners on worker safety and environmental protection.

Conversion time might be reduced by cannibalizing equipment from other plants or by employing used equipment, but the lack of integrated safety systems would probably result in serious accidents and deplete the skilled workforce needed to run the plant. According to one analyst, “Unless the plant had been designed for convertibility in the first place, the first victims of the conversion would be the production workers.”<sup>111</sup> It would also be difficult or impossible to clean out the pipes, pumps, and reactors well enough after CW-agent production to deceive an onsite inspection. For all of these reasons, it would probably be simpler to build a dedicated CW agent production facility than to convert an existing single-purpose plant.<sup>112</sup>

#### MULTIPURPOSE PLANT

A multipurpose plant would be easier to convert to production of nerve agents than a single-purpose plant. Multipurpose plants are common in the specialty chemical industry, and they are also operated by subcontractors known as “toilers” or custom producers who make small batches of chemicals for larger companies that do not want to invest in special equipment for this purpose. For this reason, multipurpose plants are designed for maximum flexibility. Process units, heat exchangers, and storage facilities are connected by extra pipes that can be linked in various configurations to manufacture several different

<sup>109</sup> Pittaway, *op. cit.*, footnote 20.

<sup>110</sup> Zapf, *op. cit.*, footnote 89, p. 292.

<sup>111</sup> Robinson, “Chemical Weapons Proliferation,” *op. cit.*, footnote 8, p. 26.

<sup>112</sup> Alan R. Pittaway, “An Approach to the Problem of Inspecting for Organophosphorus Chemical Munition Production, Transportation and Storage” (Booz-Allen Applied Research, Inc., August 1988), p. 14.

chemicals over the course of a year.<sup>113</sup> The equipment is generally designed to handle highly corrosive chemicals. Some process equipment may be kept on pallets to minimize conversion time, and quick-cleaning features and sophisticated electronic controls permit rapid rearrangement of components. Because of the complexity of a multipurpose plant, its operation requires highly skilled engineers and other experienced personnel.

Today, modern multipurpose facilities capable of short-term, small-batch production are not the norm in developing countries, where the great majority of companies produce large volumes of a few commodity chemicals. As a result, there are few multipurpose plants in the developing world that could be reconfigured. Nevertheless, the trend in the worldwide chemical industry is to build more multipurpose plants as a means of adjusting to rapid changes in production technology. Such a plant might therefore have the equipment needed for nerve-agent manufacture distributed among its various production processes.

If a multipurpose plant were designed for rapid conversion from one chemical process to another, it might be possible to switch over in a few days with little chance of being detected. Even so, a plant specifically designed for rapid conversion from commercial to CW-agent production would be costly to build (on the order of \$150 million), and would require a high level of technological know-how in plant design, engineering, and operation, and a skilled construction workforce. Design and construction would take about 4 years in most parts of the developing world.

A dual-use plant designed for rapid conversion would also require stringent cleaning measures for the final steps in the production process to prevent the contamination of commercial products—particularly pharmaceuticals—with deadly CW agents. Since seals on pumps and other material-handling equipment absorb chemicals from the production process, switching from production of one chemical to another requires removing the pumps and cleaning them off-line, a time-consuming process. In a rapidly convertible plant, however, the production line might be configured with modular pumps that could be removed quickly for cleaning and then replaced. Alternatively, two sets of pumps might be installed in parallel so that different chemicals could be produced on the same line without contaminating each other.<sup>114</sup> Nevertheless, a plant that has been specifically designed to facilitate rapid decontamination would probably be uneconomical for commercial production, and would therefore arouse suspicions on those grounds.

### | Binary Agent Production

Some analysts have argued that binary weapons might accelerate CW proliferation by making chemical weapons inherently easier and safer to manufacture, store, transport, and use.<sup>115</sup> Indeed, the relative lack of toxicity of the two precursors means that production plants require less stringent containment measures. In the 1950s, for example, the United States produced the binary precursors DF and QL in plants open to the outside air and with relatively few safety precautions. Illicit production of binaries is also more difficult to detect because the two chemical components have some legitimate commercial

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<sup>113</sup>“CMA’s Olson Unravels Intricacies of Verifying a Chemical Arms Treaty,” *Chemical and Engineering News*, vol. 67, No. 17, Apr. 24, 1989, p. 8.

<sup>114</sup>Interview with Kyle Olson, EAI Corp., 1992.

<sup>115</sup>Brad Roberts, “Technical Impediments to Proliferation and Binary Production,” *Binary Weapons: Implications of the U.S. Chemical Stockpile Modernization for Chemical Weapons Proliferation*, Report by the Congressional Research Service prepared for the Subcommittee on International Security and Scientific Affairs of the Committee on Foreign Affairs, House of Representatives, Apr. 24, 1984 (Washington DC: Government Printing Office, 1984), p. 14.

uses. A binary sarin weapon, for example, would consist of two ingredients, DF and isopropanol, which react spontaneously to form the nerve agent. Yet dichlor—the immediate precursor of DF—has legitimate commercial uses in fire retardants, insecticides, and plastics, and isopropanol (rubbing alcohol) is a common industrial chemical. Manufacture of these compounds for legitimate uses could thus be used as a cover for the illicit production of nerve agents. Finally, binary weapons make it possible to use standard logistics channels and less rigorous security measures during production and transport, and they have a relatively long shelf-life.

## I Trade-Offs

For a country seeking to develop a CW production capability, there are several major tradeoffs in the choice of proliferation pathway:

### SIMPLICITY V. VISIBILITY

A proliferant faces a tradeoff between the use of a proven and relatively simple production process for CW agents (e.g., conversion of thiodiglycol to sulfur mustard) and the need to conceal its activities by using less well known precursors or procedures, thereby complicating the production process. Thus, a proliferant must balance the need for secrecy against the efficiency and cost of production.

### SPEED V. VISIBILITY

If an aspiring proliferant faces a long-term adversary and seeks to acquire a strategic CW stockpile, it may seek to minimize visibility by investing the money and time needed to build a dedicated clandestine plant. If the threat is more immediate, however, it may choose to convert an existing commercial facility to CW agent production.

### SAFETY V. VISIBILITY

A proliferant may seek to minimize the visibility of a CW production facility by jury-rigging it from used equipment or items purchased from multiple suppliers. The lack of an integrated plant design would result in more hazardous operation, however, increasing the occupational risks to the workforce and the contamination of the environment near the plant. A reckless government might even deliberately accept a greater risk to its workforce or population in order to acquire a CW capability more quickly or covertly, particularly if it were a party to the Chemical Weapons Convention.

### SIMPLICITY V. SHELF-LIFE

The sophistication of the production technology required to manufacture agents depends on the urgency of a country's military requirements. If a country has no immediate need to use CW agents and plans to stockpile them for several years, the agents will require long shelf-life and must therefore be produced with high purity. If a country is producing nerve agents for immediate use in battle, however, it can afford to make a less pure product by eliminating the distillation step or the use of stabilizing additives.

### AUTONOMY V. EFFICIENCY

Using an immediate precursor of a CW agent is obviously more efficient than using a starting material that is several steps removed from the final product. Thus, while back-integration of precursor chemicals reduces a proliferant's dependence on outside suppliers, it also results in greater overall complexity and cost, requires more workers to operate the plant, and results in a larger production complex to conceal and to decontaminate.

# Appendix 2-A

## Techniques for the Detection and Analysis of Chemical Signatures

**T**he Chemical Weapons Convention (CWC) permits the collection and analysis of samples during onsite inspections. Although the details of the sampling process remain to be determined by a Preparatory Commission that is meeting in The Hague to negotiate the details of treaty implementation, several analytical techniques may be used to detect and monitor chemical signatures associated with the illicit production of CW agents. Such methods could be employed either during onsite inspections of declared chemical plants authorized by the CWC or for near-site monitoring from the perimeter of a facility or from an overflying aircraft. Clandestine CW production facilities would first have to be identified by intelligence assets and then subjected to a challenge inspection before chemical sampling could take place.

The future international inspectorate to be established under the CWC will require the establishment of accredited analytical laboratories that use certified testing procedures for identifying CW agents, precursors, and degradation products. During onsite visits, inspection teams will use specified instrumentation for performing in situ chemical analyses. To facilitate the

development of such agreed instrumentation and procedures, the Government of Finland has sponsored since 1973 the development of suitable analytical techniques. In recent years, this program has included a series of international “round-robin” experiments involving the analysis by laboratories in 15 countries of unknown samples spiked with nerve and mustard agents, precursors, and degradation products.<sup>1</sup>The participating countries have agreed that the presence of controlled compounds will be confirmed with at least two different instrumental methods of analysis, and that the analytical laboratories will implement stringent quality-control measures.

**The difficulty of detecting CW agent signatures is site-dependent and is a function of the sophistication of a plant’s emission-control and decontamination systems and the quality of its maintenance.** According to one analysis, the verification challenge ranges in difficulty from the relatively simple task of detecting the production of treaty-controlled chemicals in a large, single-purpose, stand-alone facility with a rudimentary emission-control system to the much harder problem of detecting telltale signatures at a facility equipped with an advanced environmental

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<sup>1</sup> The participating countries in the Third Round-Robin Test were Australia, **Canada, China**, the Czech and Slovak Federated Republic, Finland, France, Germany, **India**, the Netherlands, Norway, the Russian Federation (two labs), **Sweden**, Switzerland, the United Kingdom, and the United States (two labs). See **Marjatta Rautio**, ed., *International Interlaboratory Comparison (Round-Robin) Test for the Verification of Chemical Disarmament. F.3. Testing of Procedures on Simulated Military Facility Samples* (Helsinki: Ministry of Foreign Affairs of Finland, 1992).

control system and embedded in a large multipurpose chemical complex.<sup>2</sup>

Since the analytical techniques described below can reveal a considerable amount of information regarding the operation of a chemical facility, verifying the CWC must balance the intrusiveness needed to detect treaty violations against the risk of compromising confidential business information unrelated to the treaty.<sup>3</sup> In order to minimize this risk chemical analyses for CWC verification will not involve an exhaustive characterization of samples but will focus instead on the search for a specific set of known chemicals associated with CW production. Screening for a set of known target compounds poses less of a threat to proprietary information than would a complete chemical analysis of the sample. If one or more suspect chemicals were detected in the waste stream, however, a more in-depth analysis might be warranted.

## ONSITE INSPECTION TECHNIQUES

During the production of a CW agent, traces of various chemicals are released in vapor form from the plant's smokestacks and ventilation systems and are also absorbed by the seals and gaskets on pumps and other fittings, the agitator in the reaction vessel, and various rubber components and grease seals. Thus, during onsite inspections; of a chemical facility, inspectors might disassemble pumps and other pieces of equipment close to the production vessel, or take swipe samples from inside the machinery, which is not likely to be flushed clean by conventional decontamination methods.<sup>4</sup> In order to ensure that the samples do not degrade before being analyzed, inspectors must use proper sampling techniques (e.g., dry v. wet swipes). They must also determine whether actions have been taken to preclude access to possible samples, such as painting over a stain on the floor.

CW agents and precursors break down in the environment through the action of ultraviolet radiation (photolysis), water (hydrolysis), and air (oxidation),

resulting in a series of degradation byproducts. Environmental factors such as sunlight, weather, temperature, and soil type can influence the rate of degradation. Dilution is another key factor affecting detectability: chemicals in effluents discharged into a river, for example, may be diluted to undetectable levels a few hundred meters or so downstream from the outflow pipe.<sup>5</sup>

Determining the presence or absence of known chemicals is generally performed with some variant of a gas chromatograph/mass spectrometer (GC/MS), an instrument that combines two analytical methods in tandem. First, the gas chromatography vaporizes the sample and passes it through a packed column or a hollow glass capillary tube lined with a fine polymer material. Various substances in the sample take different amounts of time to emerge from the tube, depending on their molecular weight and their attraction to the polymer lining. As they emerge from the chromatography, constituents of the sample are then introduced into a mass spectrometer, which breaks them up into a compound-specific set of molecular fragments and then measures their masses very precisely.

Sorting molecules first by their retention time in the chromatography and then by the masses of their constituent parts, GC/MS analysis can reliably identify each of several compounds in a sample. Such identification is usually performed automatically by a pattern-recognition algorithm, which tries to match the mass spectrum of each component against a computer database containing tens of thousands of reference spectra of known chemical compounds and comes up with one or more candidates with specified probabilities. For purposes of CWC verification, considerable effort has gone into compiling "libraries" of GC/MS spectra for CW agents, precursors, and degradation products.

If the GC/MS instrument is calibrated correctly, it can confirm very reliably whether a given

<sup>2</sup> James D. Barden et al., *Remote Sensing Technology and CW Arms Control* (Alexandria, VA: Kaman Sciences Corp., Report No. P650-1254G-1, Feb. 2, 1993), p. 7.

<sup>3</sup> For an in-depth discussion of this issue, see U.S. congress, Office of Technology Assessment, *The Chemical Weapons Convention: Effects on the U.S. Chemical Industry*, O'TA-BP-ISC-106 (Washington, DC: U.S. Government Printing Office, August 1993).

<sup>4</sup> "CMA's Olson Unravels Intricacies of Verifying a Chemical Arms Treaty," *Chemical and Engineering News*, vol. 67, No. 17, Apr. 24, 1989, p. 9.

<sup>5</sup> Albert Verweij et al., "Chemical Warfare Agents: Verification of Compounds Containing the Phosphorus-Methyl Linkage in Waste Water," *Science*, vol. 204, May 11, 1979, p. 617.

**chemical is present in a sample at remarkably low concentrations, even in complex mixtures.** The device can detect substances in the parts per trillion range, although the more complicated the mixture is, the harder it is to reach such high sensitivities. If the sample is being tested for the presence of a known chemical, the detection limit will be much lower than for an unknown chemical. GC/MS is sensitive enough to detect nerve-agent degradation products in waste water even after extensive purification efforts. Empirical results also indicate that detectable traces of CW agents may persist for long periods after production. In one trial inspection, traces of a carbamate pesticide were found 2 months after production ended by analyzing wipe samples from equipment and waste samples, as well as air samples from **warehouses and packaging lines.**<sup>6</sup>

**Nevertheless, the extremely low detection thresholds achieved in the laboratory may not be possible in the field.** GC/MS may not be able to identify trace quantities of agent with a high probability in the complex environment of a multipurpose chemical plant, since other compounds unrelated to CW agent production may interfere with the analysis. In such cases, visual inspection of the plant could help pare down the list of possible candidate compounds to those it would be technically feasible to manufacture in that facility.

During an onsite inspection, special sample-preparation methods may be necessary. For example, a water-soluble chemical may have to be converted into a derivative that is volatile enough to pass through a gas chromatography. It may also be necessary to try to extract the target compounds from a more complex mixture or from an absorbent material such as concrete, although such custom extractions tend to be difficult, time-consuming, and expensive. Some nerve-agent precursors, for example, absorb tightly to concrete and are only released by strong acid treatment.

In addition to GC/MS, a gas chromatography can be combined with other types of detectors to perform

specific analytic tasks. For example, a *flame photometric detector* can identify the presence of sulfur or phosphorus in a sample with high sensitivity, while an *electron-capture detector* can identify fluorine and phosphorus-containing compounds. GC/MS can be complemented with other methods of chemical analysis. For example, *high-performance liquid chromatography (HPLC)* is useful for separating polar, nonvolatile compounds, Bioassays such as the acetylcholinesterase-inhibition test can detect nerve agents at very low concentrations through their ability to inactivate the enzyme acetylcholinesterase involved in neuromuscular transmission. Antibody-based techniques, such as the *enzyme-linked immunosorbent assay (ELISA)*, rely on the ability of monoclonal antibodies to detect trace quantities of target compounds with high sensitivity, although their specificity may be relatively poor. Monoclonal antibodies have been produced for most of the major CW agents.<sup>7</sup> Finally, research and development is under way on *biosensors*, in which binding of the target compound to specific antibodies or cellular receptor molecules triggers an optical, physical, or electrochemical change that can be converted into an electrical signal.

In the hypothetical case of a whole new class of CW agents whose spectra are not already stored in a computer database, one would have to use an analytical method that provides detailed structural information from which the identity of the molecule can be deduced. GC/MS can provide useful information about unknown compounds, such as their molecular weight and elemental composition. In addition, *nuclear magnetic resonance (NMR)* spectroscopy is often used in conjunction with other techniques such as infrared and Raman spectroscopy to derive a molecular structure for unknowns. Nevertheless, structure determination with NMR requires a fairly pure sample in the milligram range, many orders of magnitude greater than the minimum concentration at which a known compound can be detected with GC/MS.<sup>8</sup>

<sup>6</sup> Gordon M. Burck, "Chemical Weapons Production Technology and the Conversion of Civil Production," *Arms Control*, vol. 11, No. 2, September 1990, p. 141.

<sup>7</sup> C. N. Lieske et al., "Development of an Antibody that Binds Sulfur Mustard," *Proceedings of the 1991 Medical Defense Bioscience Review (Fort Detrick, MD: U.S. Army Medical Research Institute of Chemical Defense, Aug. 7-8, 1991)*, pp. 131-134.

<sup>8</sup> Manuel Sanchez et al., *Chemical Weapons Convention (CWC) Signatures Analysis (Arlington, VA: System Planning Corp., Final Technical Report No. 1396, August 1991)*, p. 23.

## Chemical Signatures

Sulfur mustard breaks down in the environment into thiodiglycol and two impurities, thioxane and dithiane, which can be identified as signatures of mustard production.<sup>9</sup> Most nerve agents (e.g., sarin, soman, and VX, but not tabun) contain a phosphorus-methyl (P-CH<sub>3</sub>) bond that is difficult to break; it remains intact after chemical treatment and can only be destroyed by aggressive treatments such as high-temperature incineration.<sup>10</sup> These nerve agents break down in the waste stream into methylphosphonate (which contains the phosphorus-methyl bond), whereas most organophosphorus pesticides are degraded to phosphoric acid. (See figure 2A-1.)

**The durability of the phosphorus-methyl bond also means that it can be identified for long periods after being discharged into the environment. For this reason, the phosphorus-methyl linkage is thus a good signature of illicit nerve-agent production.** For example, soil samples taken in late 1992 from bomb craters near a Kurdish village in northern Iraq by a team of forensic scientists and later analyzed with GC/MS were found to contain degradation products of sarin and mustard gas more than 4 years after the village was bombed by the Iraqi army in 1988. This finding suggests that traces of CW agents or their degradation products can be detected after persisting in the environment for long periods, provided that the samples are taken from a point of high initial contamination such as the center of a bomb crater.<sup>11</sup>

Chemical signatures associated with the production of CW agents could also be obtained from sampling the waste effluent stream of a production plant, although the samples would have to be collected before significant dilution occurred. At the same time, little additional data would probably be derived from

visiting the plant's control room (where the relevant information could be hidden), sampling from the production line (which might interfere with production), or examining the plant's books (which could be forged).<sup>12</sup> In order to ensure that the waste stream was actually connected to the production line, however, the inspectors would have to be given unlimited access to the plant's waste-processing system.

## I Problem of False Positives

Since GC/MS analysis is so sensitive, it is unlikely to yield "false negatives," that is, to conclude mistakenly that a sample contains no evidence of illicit production. However, the problem of "false positives"—unfounded suspicions of noncompliance—is more troublesome, particularly with respect to early precursors and final degradation products. In the case of **nerve agents, false-positives can arise if the plant is manufacturing or using a legitimate compound that contains a phosphorus-methyl bond and thus breaks down into the same degradation product as a nerve agent.** Fortunately, only a handful of commercial products contain a phosphorus-methyl bond, including the pesticide Mecarphon and the organophosphorus flame retardant dimethyl methylphosphonate (DMMP), which is also used as a plasticizer for vinyl plastic and an intermediate in the production of herbicides.

Worldwide production of DW is spread among 14 companies, 11 in the United States and 3 in Western Europe.<sup>13</sup> According to one assessment, "A challenged facility may claim it is producing a chemical closely related to a scheduled agent [CW agent or precursor] which would result in emissions overlapping those of the scheduled agent. As a result, some identified chemicals may not be sufficiently unique for

<sup>9</sup> Sanches et al., *Analysis of Signatures Associated with Noncompliance Scenarios*, Report No. DNA-TR-92-74 (Arlington, VA: Systems Planning Corp., January 1993), p. 59.

<sup>10</sup> See U.S. Congress, Office of Technology Assessment, *Disposal of Chemical Weapons: Alternative Technologies—Background Paper*, OTA-BP-O-95 (Washington, DC: U.S. Government Printing Office, June 1992).

<sup>11</sup> Human Rights Watch, "Scientific First: Soil Samples Taken From Bomb Craters in Northern Iraq Reveal Nerve Gas—Even Four Years Later," press release, Apr. 29, 1993; Lois Ember, "Chemical Weapons Residues Verify Iraqi Use on Kurds," *Chemical & Engineering News*, vol. 71, No. 18, May 3, 1993, pp. 8-9.

<sup>12</sup> Stephen Black, Benoit Morel, and Peter M. Zapf, "E Laminating the Shadows: On-site Inspections and the Chemical Weapons Convention" Benoit Morel and Kyle Olson, eds., *Shadows and Substance: The Chemical Weapons Convention* (Boulder, CO: Westview, 1993), p. 193.

<sup>13</sup> U.S. Department of Commerce, Bureau of Export Administration, Office of Foreign Availability, *Foreign Availability Review: 50 CW Precursor Chemicals (II)* (Washington DC: Department of Commerce, Nov. 8, 1991), p. 23.



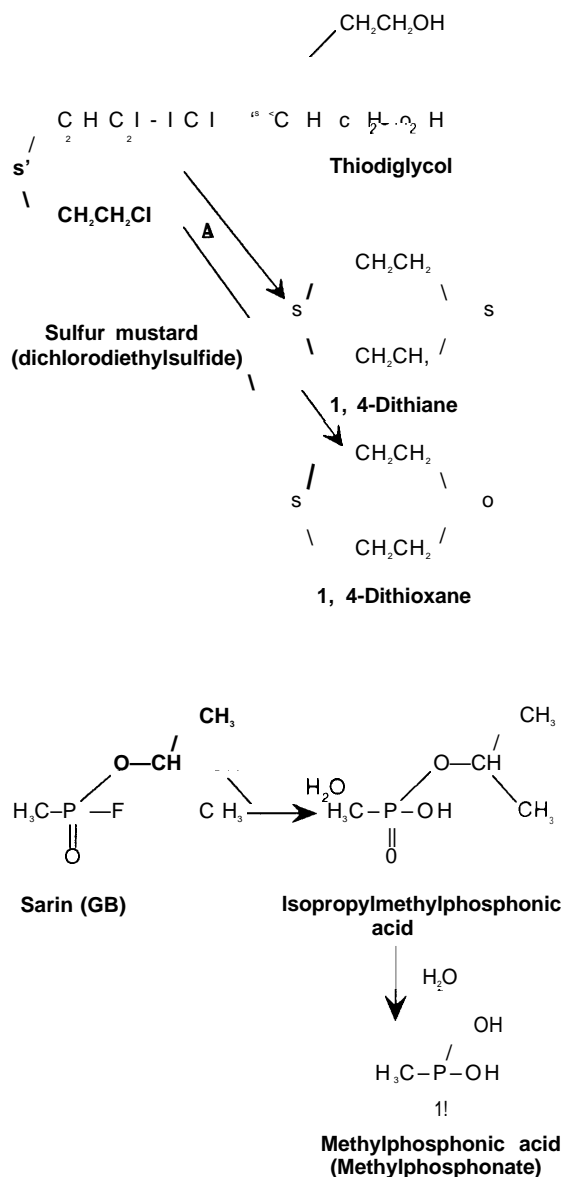
this particular plant. The same situation applies to illicit facilities embedded within larger related plants.<sup>14</sup>

Because methylphosphonate is resistant to further degradation, it tends to accumulate in the environment. Background levels have therefore been increasing gradually in the rivers, lakes, and streams of industrialized countries. For example, Albert Verweij and colleagues in the Netherlands detected significant levels of the compound in the waters of the Rhine and Meuse Rivers because of the upstream manufacture of DMMP.<sup>15</sup> Such environmental background levels may either generate false-positives (unless they had been previously measured) or, conversely, mask the actual effluents from nerve-agent production. For this reason, the detection of trace levels of methylphosphonate in the air, soil, or water near a chemical plant might not provide unequivocal evidence of nerve-agent production.

To solve the problem of false positives, it would be essential to screen liquid and gaseous chemical-plant emissions for a specific set of target compounds. In addition to the CW agents themselves and their degradation products, this list would include agent precursors and intermediate byproducts generated at various steps in the manufacturing process, and their respective degradation products. The major weaponized CW agents each have up to six different synthetic routes, requiring different sets of equipment and precursor chemicals. Thus, a suite of target compounds could provide evidence for each of these alternate production pathways. Identifying such suites of chemical compounds in the waste stream would reduce the likelihood of false-negatives and false-positives.

Detecting traces of nerve agents themselves clearly provides the best evidence of illicit production. If the actual agents cannot be found, the next best evidence is provided by primary degradation products and, if possible, both parts of the original agent molecule (e.g., the acid and amine components of VX). The detection of a secondary degradation product such as methylphosphonate would not in itself constitute

Figure 2-A-I-Chemical Warfare Agents and Degradation Products



SOURCE: *Chemical and Engineering News*, vol. 71, No. 18, May 3, 1993, p. 8.

<sup>14</sup>Sanches et al., op. cit., footnote 9, p. 65.

<sup>15</sup>Verweij et al., op. cit., footnote 5, p. 617.

strong evidence of illicit production because it **could also result from certain legitimate chemicals**.<sup>16</sup> Nevertheless, if methylphosphonate is detected in the waste stream and plant officials seek to explain it away by claiming pesticide or DMMP production, the inspectors could ask for supporting evidence in the form of samples and written records.

### | Circumvention Scenarios

In addition to the problem of false-positives, there is the possibility of deliberate deception on the part of a determined proliferant. For example, a country engaged in clandestine CW-agent production might take special measures to mask or otherwise conceal the presence of **telltale chemical signatures in the waste stream**.<sup>17</sup> Indeed, a problem associated with sensors designed to detect trace quantities of chemicals is that they can easily be swamped by related signals. Examples of some possible deception strategies include:

- Pumping chemical wastes from the plant into underground storage tanks or wells, or into tanker trucks for disposal off-site.
- Setting up a phoney waste stream for sampling that is unconnected to the actual production line.
- Continually recycling the waste stream to reduce the quantities of byproducts released.
- Using a decontaminating solution that reacts with **traces** of illicit chemicals to form a product that may not be in the standard library of a GC/MS. This strategy has been termed “designer decontamination.”<sup>18</sup> For example, the methyl phosphonate in the waste stream could be reacted with thionyl chloride and an alcohol to obtain a diester, which would not look anything like the original compound in a GC/MS analysis. Nevertheless, the use of an unusual decontaminating solution would be suspicious if nothing about the facility justified its presence; in addition, the sample could be hydrolyzed during the analysis to regenerate the methylphosphonate.

- Diluting the release of a telltale byproduct such as methylphosphonate in the waste stream so that it can no longer be detected. In practice, the effectiveness of this strategy would depend on the detection limits of the analytical instrument, which for GC/MS can reach parts per trillion. Thus, achieving the necessarily dilution to evade detection would require impractically large volumes of decontamination fluid.
- Flushing the production line with a decontaminating solution followed by a legitimate but closely related commercial product to mask any residues of agent. This scheme would only be possible if the plant were simultaneously producing a commercial compound containing a phosphorus-methyl bond, such as an alkylated pesticide (e.g., methyl-parathion) or DMMP. A sophisticated cheater, however, would almost certainly couple the two operations.
- Passing production wastes through an ion-exchange resin to remove methylphosphonate; such resins are expensive but reusable.
- Developing a novel agent that is not in the GC/MS **database**. Russian scientists, for example, have reportedly developed a new type of binary nerve agent.<sup>19</sup> Such a scenario is unlikely in most developing countries, however, since the development of an entirely new class of CW agents would require a costly investment in research, development, and testing. Modifications of existing CW agents might be detected by programming the instrument’s computer to recognize a family of related agents.

Some of these circumvention strategies might reduce the probability of detection. If, however, they were performed after notification of a challenge inspection, they might be carried out hastily and carelessly, resulting in spills or other accidents that would leave behind telltale traces of agent. The use of unusual decontamination strategies might also raise suspicions of a violation. Thus, such waste effluent

<sup>16</sup> Marjatta Rautio, ed., *International Inter-Laboratory Comparison (Round-Robin) Test for the Verification of Chemical Disarmament. F.1. Testing of Existing Procedures* (Helsinki: Ministry of Foreign Affairs of Finland, 1990), p. 93.

<sup>17</sup> See Kathleen C. Bailey, “Problems With a Chemical Weapons Ban,” *Orbis*, vol. 36, No. 2, spring 1992, pp. 239-251.

<sup>18</sup> *Ibid.*, p. 241.

<sup>19</sup> “Mirzayanov, Fedorov Detail Russian CW Production,” *Novoye Vremya*, No. 44, October 1992, pp. 4-9 (translated in FBIS-SOV-92-213, Nov. 3, 1992, pp. 2-7),

sampling techniques would be most effective when used in conjunction with other forms of onsite inspection.

Searching for a suite of compounds (agents, precursors, and degradation products) on a target list would also help defeat circumvention efforts, since the *pattern* of chemical signatures emitted by a plant could not be masked as easily as a single chemical. Indeed, the likelihood of masking all of the target compounds associated with a given production process would be very low. Conversely, **whereas a secondary degradation product like methylphosphonate may give rise to false-positives, the probability of an error declines rapidly when the suspect chemical is found in conjunction with a suite of other target compounds in the same manufacturing process.**

**In sum, it remains an open question whether a carefully planned and executed deception aimed at illicit production of CW agents would be detected. Nevertheless, cheaters would probably not be sure they could get away with the deception, and hence might be deterred from trying. While one might theoretically conceive of a plant design that could circumvent detection, such a facility would probably differ significantly from existing commercial plants and might therefore arouse suspicion on those grounds.** According to one analysis:

In a multipurpose plant. . . industry would invest significantly so that the interior of the actual production line could be easily cleaned in order to enable quick product change; this would not be the case for waste water channels, reactor ventilation systems, off-specification lines and so on, which would be connected either to purification stations or to equipment used to recycle certain chemicals. It is there inspections would look for traces of illicit production; if they were designed in such a way that they could easily and thoroughly be decontaminated, this would be an

economically unfeasible and suspect effort by civil industry.<sup>20</sup>

## NEAR-SITE AND REMOTE MONITORING TECHNIQUES

Near-site and remote monitoring of chemical signatures will probably be carried out openly within the negotiated terms of the Chemical Weapons Convention and covertly as an intelligence operation. Covert sensors, by definition, could not be openly discussed they would have to be made sufficiently reliable and rugged to permit long periods of unattended operation in a potentially hostile environment and cleverly disguised to prevent detection and tampering.<sup>21</sup>

Near-site monitoring can be either *real-time*, meaning that the concentration of a particular substance is monitored continuously, or *integrative*, meaning that only the average or the cumulative amount over a period of time is recorded. Integrative monitoring can be further subdivided into *active* and *passive* methodologies. Active-integrative systems pump air or water through a filter over a period of days or weeks to concentrate trace molecules for later analysis. In contrast, passive-integrative systems simply absorb and retain trace chemicals from the environment over a period of time, much like a sponge.

### I Air-Sampling Systems

Active air-sampling could be conducted either with a system on the ground in the vicinity of a chemical plant, or based on an overflying aircraft.<sup>22</sup> **There are two types of gaseous emissions from a CW production plant: controlled smokestack emissions and “fugitive” emissions.** Stack emissions are planned releases from the production process that have been filtered by the plant’s pollution-control system. Fugitive emissions are uncontrolled releases that have not passed through the pollution-control system, such as slow leaks from storage tanks, gaskets, and reactor pressure-release valves, or an accidental production-

m J. Perry Robinson and Ralf Trapp, “Production and Chemistry Of Mustard Gas,” S. J. Lundin, ed., *Verification of Dual-use Chemicals under the Chemical Weapons Convention: The Case of Thiodiglycol*, SIPRI Chemical & Biological Warfare Studies No. 13 (Oxford, England: Oxford University Press, 1991), p. 15.

<sup>21</sup> Franklin E. Walker, *Technical Means of Verifying Chemical Weapons Arms-Control Agreements* (Washington, DC: Foreign Policy Institute, Johns Hopkins University School of Advanced International Studies, May 1987), p.15.

<sup>22</sup> Ministry for Foreign Affairs of Finland, *Air Monitoring as a Means for Verification of Chemical Disarmament*, vols. I-III (Helsinki: Ministry for Foreign Affairs, 1985-1987).

line rupture. Since the chemicals involved in CW production are not particularly volatile, fugitive emissions would tend to stay closer to the ground, and might be detected through real-time sampling at locations near the production equipment. Moreover, fugitive emissions (e.g., from storage tanks or the waste-treatment system) may persist even after a plant has been temporarily shut down.

Air sampling involves collecting samples of air downwind from a chemical facility and analyzing them for CW agents, precursors, or byproducts; it can be either real-time or integrative. In one approach, atmospheric contaminants could be pumped through a tube packed with an absorbing substance (e.g., resin beads), concentrated for a period of time, and later driven off by flash heating in an inert-gas atmosphere and identified with GC/MS or high-performance liquid chromatography. Air-borne chemicals may also adhere to dust particles or and maybe transported in droplets of water vapor, raindrops, or snowflakes.<sup>23</sup> In an experiment conducted in Finland, 4 kilograms of a nerve-agent simulant containing phosphorus were released into the atmosphere and subsequently identified in air samples collected 200 kilometers from the release site.<sup>24</sup> The U.S. Army is also developing atmospheric monitoring systems to protect the public from accidental leaks during the destruction of CW agent stockpiles.

In the treaty-verification context, detection sensitivities for air sampling are demanding for the following reasons:

- Given the lethality of CW agents, production plants usually incorporate high-containment features that minimize emissions. The more modern the plant design, the lower the level of fugitive emissions and the more difficult detection becomes. Developing countries tend to impose less stringent safety practices, but the extent of fugitive emissions varies greatly from plant to plant. The trace amounts released into the atmosphere might not be concentrated enough to create an identifiable signal.

- The majority of materials involved in CW agent production that yield detectable signatures are not very volatile even if a leak occurs, compounding the sensitivity problem.
- When air samples are taken over longer ranges, weather patterns can complicate efforts to identify the source of detected emissions, since wind may shift the direction of the emission plume. Remote air sampling cannot pinpoint the source of a clandestine facility for challenge inspection unless the sampling is conducted for extensive periods or happens to coincide with the release of detectable emissions, and unless an atmospheric-transport model can trace the contaminants back to the facility of origin.
- Deliberate countermeasures might foil air-sampling efforts. A clever plant operator might be able to mask such releases, particularly if he had prior knowledge of the monitoring technologies. Alternatively, a cheater who was aware he was being monitored might control emissions or discontinue production while samples were being collected, or refuse permission for aerial overflights. Although fugitive emissions (e.g., from storage tanks) might continue in the absence of production, they might not be concentrated enough to be detectable.

Because of these factors, **even in those instances where detection has been accomplished by air sampling, the detection was made through extensive sampling grids during rather massive releases. This source is easy to extinguish simply by stopping production.**<sup>25</sup>

## | Optical Detection Systems

Another approach to the real-time detection and analysis of chemicals released deliberately or accidentally from a CW production facility is to use a remote spectroscopic system based on light scattering, absorption, or induced fluorescence. A combination of two or more of these techniques may be needed to produce

<sup>23</sup> Amy Smithson and Michael Krepon, *Strengthening the Chemical Weapons Convention Through Aerial Inspections*, Occasional Paper No. 4 (Washington, DC: Henry L. Stimson Center, April 1991), p. 13.

<sup>24</sup> Ministry for Foreign Affairs of Finland, *The Finnish Research Project on the Verification of Chemical Disarmament* (Helsinki: Ministry for Foreign Affairs, 1989), p. 13.

<sup>25</sup> Raymond R. McGuire, *Treaty Verification Program*, Lawrence Livermore National Laboratory, personal communication, 1992.

reliable results.<sup>26</sup> Remote spectroscopy can either be “passive,” which analyzes electromagnetic radiation emitted by the sample or by background sources, or “active,” which irradiates the sample with a laser beam. For example, *fourier transform infrared spectroscopy (FTIR)* has been used to detect telltale chemical signatures in stack plumes or fugitive emissions at ground level or at higher altitudes. Broadband infrared has the potential to identify a wide variety of compounds simultaneously.

A closely related active laser sensing technique is known as lidar, for “light detection and ranging.” Whereas spectrometers are generally broad-band techniques, lidar is laser-based and thus consists of a single or a few distinct wavelengths. (As lasers become tunable, however, this distinction may disappear). An advantage of laser-based systems is that the power is focused at a single wavelength rather than being spread among many. *Differential absorption lidar (DIAL)* uses two different wavelengths, one of which is absorbed by the target molecule and one that is not. The difference between the absorbed and unabsorbed signals is used to determine the target molecule’s concentration. Another lidar technique, known as *Raman spectroscopy*, involves exciting a chemical with a monochromatic laser and measuring shifts in frequency that provide structural information. Water is not a strong Raman absorber and thus causes little interference.

Remote sensing of chemical-plant emissions maybe performed on stack plumes or fugitive emissions at ground level or at higher altitudes. In principle, the illuminating laser can be located on the ground or mounted on an aircraft, a remotely piloted vehicle, or even a satellite. To characterize the chemical emissions from a smokestack, the laser would be pointed either directly at the gaseous exhaust emitted from the stack or downwind along the effluent plume, and the returned light picked up by a detector. Fluorescence or

absorption of light by the chemical compounds in the exhaust give rise to characteristic spectral bands.

“Closed-end” optical detection systems employ a mirror or separate detector to analyze the illuminating laser beam after it passes through the chemical plume. They are more sensitive than “open-ended” systems, which collect only light scattered back to a detector near the laser source. The FTIR detector, under development for the Environmental Protection Agency, is an example of a closed-end system. It emits a beam of infrared light across the plume, and a large mirror then reflects the beam back to the emitter/detector system, doubling the path length and thereby increasing the sensitivity. After being processed the resulting data yield the characteristic infrared absorption spectra for the chemical species of interest.<sup>27</sup>

The success of optical remote-sensing techniques depends on a number of variables, however, including:

- the concentration of the target compound(s) in the plant emissions, which may be a function of emission controls;
- the chemicals present in the background and their concentrations;
- the detection limits of the remote-sensing equipment.

Current-generation systems are not sufficiently sensitive to **detect trace quantities of agent**. For example, lidar technologies are capable of detecting CW agent in air at concentrations of 1 to 10 milligrams per cubic meter. In other words, they are several orders of magnitude less sensitive than existing analytical instruments used for onsite sampling, which have a detection limit of 1 to 10 micrograms per cubic meter.<sup>28</sup> Experience has shown that the probability of remotely detecting activities occurring within a manufacturing facility is nearly zero if samples are collected more than a few meters from the building. Waste effluent streams are an exception to this rule, but even here samples must be collected before significant

<sup>26</sup> Kenneth E. Apt, Los Alamos National Laboratory, “Near-Site Monitoring for Compliance Assessment of the Chemical Weapons Convention” LACP-90-289, June 15, 1990.

<sup>27</sup> Robert Lentz et al., *Chemical Weapons (CW) Treaty Verification Technology Research and Development: program Interim Summary* (Aberdeen Proving Ground, MD: Chemical Research, Development & Engineering Center, Report No. CRDEC-CR-124, September 1991), p. 24.

<sup>28</sup> Manuel L. Sanches, et al., *Analysis of Signatures Associated With Noncompliance Scenarios*, Report No. DNA-TR-92-74 (Arlington, VA: System Planning Corp., January 1993), p. 96.

dilution occurs.<sup>29</sup> Moreover, chemical plants in developing countries do not now employ sophisticated environmental protection devices, but as such equipment becomes more widely available, plant emissions could be reduced significantly.

Potential countermeasures also exist to remote-sensing technologies. For example, a determined cheater might reduce emissions below the detection threshold, or release masking compounds that absorb infrared radiation at the same frequencies as do the target chemical species.

### | Sorbent Materials

One approach to passive-integrative monitoring involves the use of absorbent materials called “sorbents,” which have a very large internal surface area. Airborne chemicals simply diffuse into the material and are irreversibly bound to it, although they can later be extracted for chemical analysis. Examples of sorbent materials include *diatoms* (porous, silica-based structures that are the microscopic skeletons of plankton), *zeolites* (long-chain polymers of silicon, oxygen, and aluminum), and *silica gels* that have been chemically modified to absorb organic chemicals but not water.

Conceivably, artificial rocks or gravel made of a sorbent material could be dispersed in the vicinity of a suspect facility. These sorbents would accumulate volatile chemicals from the air over an extended period of time, providing concentrated samples for laboratory analysis.<sup>30</sup> The drawbacks of passive-integrative systems are the lack of temporal information about the timing of effluent releases, plus the fact that chemical agents may degrade in the natural environment or within the absorbent material.

### BIOMARKERS FOR CW AGENTS

Wartime or occupational exposure to CW agents can leave behind long-lasting traces in humans or other living organisms. These biochemical signatures, known as “biomarkers,” might conceivably be monitored as a means of detecting illicit CW production or use. During the Iran-Iraq War, for example, chemical analysis of urine samples from Iranian soldiers attacked with sulfur mustard revealed elevated levels of the metabolize thiodiglycol in most of the victims. In some cases, however, the technique could not distinguish between control urines and samples of allegedly exposed soldiers. To solve this problem, scientists at the U.S. Army Medical Research Institute of Chemical Defense developed a more sensitive assay that involved chemically derivatizing thiodiglycol before conducting the analysis.<sup>31</sup> Using this method, levels of urinary thiodiglycol in individuals moderately exposed to mustard gas were found to be greater than 10 nanograms per milliliter (10 parts per billion) for at least a week.<sup>32</sup> Similar techniques have been developed for detecting the major metabolizes of nerve agents (methylphosphonate esters) in biological fluids by converting them into derivatives suitable for gas-chromatographic analysis.<sup>33</sup> The advantage of urinary metabolizes is that measuring them is much less invasive than taking blood samples; the drawback is that most organophosphorus compounds are cleared from the body within 48 hours of exposure.

Another biomarker technique involves measuring the activity of the enzyme acetylcholinesterase, which is specifically inhibited by nerve agents. While this enzyme is located primarily in nervous tissue, it is also present in the blood—both plasma and red blood

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<sup>29</sup> Raymond R. McGuire, *op. cit.*, footnote 25.

<sup>30</sup> w. Earl, Los Alamos National Laboratory. “Specialized Sorbents,” presentation at the Chemical Weapons Convention Verification Technology Research and Development Conference, Herndon, VA, Mar. 3, 1993.

<sup>31</sup> E. M. Jakubowski et al., “Quantification of Thiodiglycol in Urine by Electron Ionization Gas Chromatography-Mass Spectrometry,” *Journal of Chromatography, Biomedical Applications*, vol. 528, 1990, pp. 184-190.

<sup>32</sup> E. M. Jakubowski et al., “Case Studies of Accidental Human Mustard Gas Exposure: Verification and Quantification By Monitoring Thiodiglycol Levels,” *Proceedings of the 1991 Medical Defense Bioscience Review (Fort Detrick, MD: U.S. Army Medical Research Institute of Chemical Defense, Aug. 7-8, 1991)*, pp. 75-80.

<sup>33</sup> M. L. Shih et al., “Detection of Metabolizes of Toxic Alkylmethylphosphonates in Biological Samples,” *Biological Mass Spectrometry*, vol. 20, 1991, pp. 717-723.

cells—although its function there is unknown. It is possible to measure the activity of blood acetylcholinesterase compared with known normal values (preferably with earlier values from the same person or a set of normal values from several individuals); the effects of nerve-agent exposure on the activity of the enzyme are detectable for up to 3 weeks.<sup>34</sup> Measurements can be made on small blood samples drawn from the fingertip. This technique has been used for routine health control of workers involved in production or spraying of organophosphorus pesticides, and it might also reveal the clandestine production of nerve agents at a suspect production or storage facility. Nevertheless, the assay would not be able to distinguish between the illicit production of nerve agents and the legitimate production of organophosphorus pesticides or fire retardants in the same plant. It would also be essential to know the background (pre-exposure) levels of acetylcholinesterase activity.

**Yet another means of detecting exposure to CW agents involves the detection of “adducts” resulting from the binding of toxic chemicals directly to molecules of DNA or protein in the body. Sulfur mustard, for example, forms covalent bonds with nucleotide bases along the DNA strand that may persist for several days or weeks. The major DNA adduct produced by sulfur mustard is an alkyl group bound to the nucleotide guanine, which accounts for over 60 percent of the DNA damage caused by sulfur mustard.<sup>35</sup> The DNA molecules can be extracted from skin cells or peripheral white blood cells and analyzed. A group of Dutch scientists has also developed monoclonal antibodies to alkylated guanine, making it possible to use an immunoassay (ELISA) technique to detect adducts in DNA extracted from white blood**

cells. This method is sensitive enough to detect one DNA adduct among  $10^8$  unmodified nucleotides—a level of damage resulting from exposure to a small dose of sulfur mustard.<sup>36</sup>

Analysis of DNA adducts can reveal an individual’s prior exposure to toxic chemicals, and has already been used to monitor occupational exposure to pesticides through both the air and the skin. This technique might also be used to detect clandestine production of CW agents by plant workers at suspect facilities, although there may be constitutional barriers to mandatory blood testing in some countries. Monitoring of DNA adducts also has some technical drawbacks. Only small quantities of adducts can be extracted from accessible tissue such as white blood cells, and DNA adducts tend to be removed by chemical and enzymatic processes and hence do not persist for long in the body, having a half-life of a few weeks.

Because of the transience of DNA adducts, several investigators have turned instead to protein adducts, **such as the alkylation of hemoglobin by sulfur mustard.** Experiments have shown that about 1,000 times more sulfur mustard binds to proteins than to DNA.<sup>37</sup> Moreover, hemoglobin has a relatively long lifespan (120 days), permitting the determination of cumulative exposure to toxic chemicals over a period of months.<sup>38</sup> Analysis of blood samples for hemoglobin adducts might therefore be the best way of detecting long-term exposure to CW agents in chemical-plant workers. Nevertheless, the concentrations of hemoglobin adducts are usually found at extremely low levels (femtomoles or picomoles per gram), requiring measures that are extremely sensitive and selective.<sup>39</sup> Such testing therefore entails complex tradeoffs among sensitivity, specificity, and cost.

<sup>34</sup> S. J. Lundin, “The Inhibition of Cholinesterase Activity by Organophosphorus Compounds as a Means in an Inspection Procedure,” in Stockholm International Peace Research Institute, *The Problem of Chemical and Biological Warfare, Vol. VI: Technical Aspects of Early Warning and Verification* (Stockholm: Almqvist & Wiksell, 1975), pp. 180-181.

<sup>35</sup> David B. Ludlum, Paula A. Ritchie, and Miasnig Hagopian, “Systemic Toxicity of Sulfur Mustard: A Predictive Test Based on the Measurement of DNA Adduct Formation in Peripheral Blood,” *Proceedings of the 1991 Medical Defense Bioscience Review (Fort Detrick, MD: U.S. Army Medical Research Institute of Chemical Defense, Aug. 7-8, 1991)*, pp. 97-100.

<sup>36</sup> H. P. Benschop et al., “Immunochemical Diagnosis and Dosimetry of Exposure to Sulfur Mustard,” *Proceedings of the 1991 Medical Defense Bioscience Review (Fort Detrick, MD: U.S. Army Medical Research Institute of Chemical Defense, Aug. 7-8, 1991)*, pp. 67-74.

<sup>37</sup> *Ibid.*, p. 72.

<sup>38</sup> Gary T. Vaughan and T. Mark Florence, “Biomonitoring of DNA-Damaging Toxins,” Dianne Watters et al., eds., *Toxins and Targets: Effects of Natural and Synthetic Poisons on Living Cells and Fragile Ecosystems* (Philadelphia PA: Harwood Academic Publishers, 1992), p. 172.

<sup>39</sup> *Ibid.*, p. 172.