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B R I E F

Chemical weapons detection: inspecting Syria

David Cliff, Russell Moul and Ariane Jugieux

“The use of chemical weapons has a long history, though today the use of such weapons is seen to violate a well-established norm observed in almost all quarters of the world.”

1. PA. D'Agostino and C.L. Chenier, 2006. 'Analysis of Chemical Warfare Agents: General Overview, LC-MS Review, In-House LC-ESI-MS Methods and Open Literature Bibliography'. Available at: <https://www.fas.org/irp/threat/cbw/drdc2006.pdf>, p. 2
2. Michael L. Moodie, 1999. 'The Chemical Weapons Threat', in: Sidney D. Drell, A.D. Sofaer and G.D. Wilson (ed.), 1999. *The New Terror: Facing the Threat of Biological and Chemical Weapons* (Hoover Institution Press: Stanford), p. 3
3. Pulmonary (Choking) Agents: Fact Sheet, from the UPMC Center for Health Security. Available at: http://www.upmchealthsecurity.org/website/our_work/biological-threats-and-epidemics/fact_sheets/choking_agents.pdf
4. Moodie, 1999, p. 3

Introduction

Over the past week, the issue of chemical weapons has sprung to the forefront of international consciousness, and to a level of prominence not seen for decades. The cause is the ongoing situation in Syria, where it is alleged that these weapons have been used on multiple occasions over the course of this year, including a major incident of suspected use in a suburb of Damascus on 21 August that may yet prompt Western military intervention in the conflict.

The use of chemical weapons in warfare has a long history, though today—and for many decades—the use of such weapons is seen to violate a well-established norm observed in almost all quarters of the world. Although a variety of treaties existed prior to the first world war, it was in the aftermath of that conflict—during which chemical weapons were used on a mass scale—that the landmark 1925 Geneva Protocol for the Prohibition of the Use in War of Asphyxiating, Poisonous or Other Gases, and of Bacteriological Methods of Warfare was concluded.

In 1993, the Geneva Protocol—which prohibited only the *use* of chemical weapons—was supplemented by the Chemical Weapons Convention (the CWC, in force since 1997), which bans the development, production, acquisition, stockpiling *and* use of chemical weapons. To oversee the implementation of the CWC, the Organisation for the Prohibition of Chemical Weapons (OPCW) was established, which continues to work to verify the destruction of chemical weapons stocks by CWC parties as well as the peaceful use of toxic chemicals in order to prevent new weapons from emerging.

Syria is one of only seven states that are not party to the CWC, which has 189 states parties in all. It is, however, party to the Geneva Protocol.

The situation in Syria today

In recent days a team of United Nations inspectors has deployed to Syria to investigate various claims that chemical weapons have been used in recent months in the civil war between the Syrian government

and the rebel groups opposed to it. The team's deployment had been delayed for many months, their mission having been set in motion back in March this year in response to a request from the Syrian government that the UN investigate the possible use of chemical weapons in the Khan al-Assal region of Aleppo province.

Since then, several more incidents of possible chemical weapons use were reported to the UN by various Western governments, prompting efforts by the UN to ensure its personnel could investigate a number of additional allegations as well. As a result, the deployment of the team was stalled until mid-August when, after several months of negotiations, it was agreed that in addition to Khan al-Assal, two other locations could be visited. The mandate in all cases was for inspectors to attempt to ascertain whether or not chemical agents had been used, but not to apportion blame for their use to either the government or opposition forces.

On 21 August, with these inspectors already in the country, reports emerged of an apparent chemical weapons attack on a massive scale in the Ghouta district of eastern Damascus. Initial media reports—accompanied by harrowing footage of evidently sick and distressed adults and children, and bodies—indicated that over a thousand may have been affected. In the wake of the attack, Medecins Sans Frontieres (MSF), a medical charity, said that three MSF-supported hospitals in Damascus treated around 3,600 patients displaying what it called 'neurotoxic symptoms' (including convulsions and breathing difficulties). Of these, it said that 355 had died.

UN inspectors travelled to Ghouta after a period of several days, once the Syrian government finally permitted them to do so (having initially refused, in what Western states called an attempt to allow evidence to degrade). Speaking on 27 August, White House spokesman Jay Carney told reporters that there was 'very little doubt in [America's] mind that the Syrian regime is culpable,' echoing comments earlier that day from US Secretary of State John Kerry, who called the use of chemical weapons in Ghouta by the Syrian

regime 'undeniable'. As this brief is being prepared, the prospect of Western military intervention continues to hover over the crisis in response to the atrocity.

Meanwhile, the UN's inspectors continue to gather what evidence they can from the site, which has now taken on far greater significance than the other three locations that the inspectors were due to visit. Against this backdrop, this brief addresses the kind of techniques that can be used in the identification of chemical warfare agents, both in the immediate aftermath or later stages after an attack, and some time after, and the complexities involved. It also, in the next section, seeks to explain the work of the inspectors currently in Syria within the context of the UN secretary-general's 'Mechanism for Investigation of Alleged Use of Chemical and Biological Weapons', under the auspices of which they are operating.

The UN secretary-general's investigative mechanism

The UN secretary-general's so-called Mechanism for Investigation of Alleged Use of Chemical and Biological Weapons is a procedure that can be initiated at the request of any UN member state. In March, the mechanism was set in motion by Syria itself, with UN Secretary-General Ban Ki-moon noting then that the UN would be operating pursuant to General Assembly resolution 42/37 C, of 1987, and Security Council resolution 620, of 1988.

Specifically, General Assembly resolution 42/37 C provides for UN member states to report possible uses of chemical or biological weapons to the secretary-general for investigation. That resolution built on another—resolution 37/98 D of 1982—which deals with the establishment of 'provisional' procedures 'to make possible the prompt and impartial investigation' of information concerning possible violations of the Geneva Protocol. For its part, Security Council resolution 620 encourages the secretary-general to carry out prompt investigations in response to any allegations of this nature by member states.

Announcing the decision to embark

on an investigation earlier this year, Mr Ban noted that UN officials were working with both the Organisation for the Prohibition of Chemical Weapons and the World Health Organization (WHO). The OPCW shall cooperate with the secretary-general in cases of alleged use involving non-parties to the Chemical Weapons Convention, like Syria (as set out in part XI, paragraph 27 of the convention's annex on verification). Cooperation between the secretary-general and the WHO in cases of alleged use is provided for under a 2011 memorandum of understanding between the two. (Incidentally, the procedure for investigating alleged use in a state party to the CWC would follow a different mechanism, as laid out in the convention's annex on verification, part XI, paragraphs 1-26.)

The next section of this brief goes on to look at the various kinds of chemical weapons, before turning to what methods can be used to detect them in cases of possible use.

Chemical warfare agents

Chemical warfare agents are classified on the basis of their effect on the human body and categorised into choking, blister, blood, and nerve agents.¹ The table on the next page lists some of their main features.

Choking agents

Choking agents, sometimes known as pulmonary agents, are chemical agents designed to impede a victim's ability to breathe. They operate by attacking lung tissue and causing a build-up of fluid in the lungs (a condition known as pulmonary oedema) when inhaled. They were the first chemical weapons used during the World War I and are thought to have been responsible for 80 percent of deaths from chemical weapons in that conflict.² These agents can be made from a wide array of chemicals, the most common of which are chlorine, ammonia and phosgene.³ These chemicals have widespread commercial use and are produced on a massive scale across the world each year.⁴

Blister agents

Blistering agents, also known as vesic-

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5. Blister Agents: Fact Sheet, OPCW. Available at: <http://www.opcw.org/about-chemical-weapons/types-of-chemical-agent/blister-agents/>
6. Ibid.
7. Blood Agents: Fact Sheet, OPCW. Available at: <http://www.opcw.org/about-chemical-weapons/types-of-chemical-agent/blood-agents/>
8. Moodie, 1999, p. 4
9. Ibid., p. 9
10. Ibid., pp. 5-38
11. <http://emedicine.medscape.com/article/831760-overview>

“The toxic effects of all chemical warfare agents depend on the concentration of the agent and the route and length of exposure.”

	Physical States	Persistency	Delivery	Route	Target
Blister Agents	Liquid, solid	High	Vapour, aerosol, liquid	Lungs, eyes, skin	Humans, animals
Blood Agents	Liquid, vapours	Low	Vapour	Lungs	Humans, animals
Choking Agents	Liquid	Low	Vapour	Lungs, eyes, skin	Humans, animals
Nerve Agents	Liquid	Low to high	Vapour, aerosol, liquid	Lungs, eyes, skin	Humans, animals

Source: adapted from Michael L. Moodie, 1999. ‘The Chemical Weapons Threat’, in: Sidney D. Drell, A.D. Sofaer and G.D. Wilson (ed.), 1999. *The New Terror: Facing the Threat of Biological and Chemical Weapons* (Hoover Institution Press: Stanford), p. 3

cants, include sulphur mustard (H, HD), nitrogen mustards (HN1, HN2, HN3), phosgene oxime (CX), and Lewisites (L1, L2, L3). They consist of oily substances that act—initially as an irritant, causing blisters, and then as a cell poison—via inhalation and through contact with the skin, eyes and mucus membranes.⁵ Blister agents were first used in combat in 1917 by Germany, and they were used widely in the Iran-Iraq War (1980-88).⁶

Blood agents

Blood agents are distributed through the body via blood. The two most known blood agents are hydrogen cyanide (AC) and cyanogen chloride (CK), which interfere with the ability of blood cells to utilise oxygen.⁷ Cyanide-based agents were used in World War I, but they were largely unsuccessful because of the large amounts needed to saturate a given space, and the fact that in vapor form it is difficult to maintain a lethal concentration. Like chlorine, cyanide compounds are produced in vast quantities for commercial purposes.⁸

Nerve agents

Nerve agents, categorised either as G- or V-series agents (which are code names, allocated when they were first produced in Germany and the UK), affect the transmission of nerve impulses by inhibiting the functioning of the vital enzyme acetylcholinesterase (AChE), which deactivates the neurotransmitter preventing continuous nerve-firing.⁹

G-series nerve agents were first discov-

ered in 1936 while researching organophosphorous pesticides. The first compounds to be developed were tabun (GA), sarin (GB), and soman (GD). G-series agents can be 100 to 1000 times more poisonous than pesticides. They act rapidly, and generally enter the body through inhalation or through the skin.

V-series agents were originally discovered in 1948 by British scientists also involved in pesticide research. Both the US and the USSR investigated military development in the 1960s.¹⁰ The V-series agents, such as ethyl S-2-diisopropylaminoethyl methylphosphonothioate (VX) and isobutyl S-2-diethylamino ethyl methylphosphonothioate (VR), are known as ‘persistent agents’, which means they can remain on skin, clothing, and other surfaces for long periods after use. The physical chemical properties of V-series agents differ from those of the G-series: their consistency is similar to oil; and so the risk of inhalation is less serious than the G-series. Their toxicity is thus linked to dermal exposure and they are approximately ten times more poisonous than sarin.¹¹

Symptoms associated with nerve agents

The toxic effects of all chemical warfare agents depend on the concentration of the agent and the route and length of exposure. According to the OPCW, characteristic symptoms of low-level exposure to nerve agents, resulting in minor poisoning, typically involves increased salivation, running nose and a feeling of pressure on the chest. The pupils contract

12. OPCW fact sheet ‘Nerve Agents’. Available at: <http://www.opcw.org/about-chemical-weapons/types-of-chemical-agent/nerve-agents/#c4116>

13. Ibid.

14. Luginbuhl and Aja Montes (2011), *Detection and Identification of Chemical Warfare Agents in Environmental Samples*, ETH Zurich, www.zenobi.ethz.ch/Analytik_2012/CWA%20Presentation.pptx

15. OSHA Technical Manual (OTM), Section II, Chapter 3 (2008), Occupational Safety and Health Administration. Available: [https://www.osha.gov/dts/osta/otm/otm_ii/otm_ii_3.html]

16. Organisation for the Prohibition of Chemical Weapons (OPCW), *Detection of Chemical Weapons*. Available: [<http://www.opcw.org/our-work/assistance-and-protection/protection-against-chemical-weapons/detection/>]

(miosis) which impairs vision and causes pain when the victim attempts to focus on nearby objects. These symptoms can be accompanied by headaches, tiredness, slurred speech, hallucinations and nausea.¹²

Higher exposure can lead to more severe symptoms. Bronchoconstriction and secretion of mucous in the respiratory system leads to difficulty in breathing and to pronounced coughing. Discomfort in the gastrointestinal tract may develop into cramps and vomiting, while the victim experiences involuntary discharge of urine and defecation. Symptoms associated with skeletal muscles are very typical, and even moderate poisoning can manifest as muscular weakness, localised tremors and convulsions.

Muscular paralysis caused by exposure to nerve agents can also affect the respiratory centre of the central nervous system and the respirator muscles. The combination of these two effects is the direct cause of death in a victim—a death caused by nerve agents is a form of suffocation.¹³

Detection procedure

In accordance with the Chemical Weapons Convention, samples can be analysed on-site by the OPCW inspection team, and, as is to be the case with Syria, transferred off-site to laboratories that have been certified for such analysis by the director-general. These inspections are intended to provide factual evidence for the presence of prohibited chemicals and/or to support a conclusion of their absence.

Field detection techniques

An array of field detection techniques are available for first responders in situations in which chemical weapons are suspected to have been used. Methods typically involve portable instruments and highlight blister agents and nerve agents as well as other toxic substances.¹⁴ Although there are multiple ways of detecting the presence and nature of chemical warfare agents, the main techniques are colorimetric detection, Ion-Mobilisation Spectrometry (IMS), flame photometry and Surface Acoustic Wave (SAW). Detectors are generally reliable, although exhibit a

tendency to read false positives and can be affected by external conditions such as temperature, humidity, and pressure.

Most methods are able to detect concentration levels below Immediate Danger to Life and Health (IDLH) however they cannot usually read levels as low as the Acceptable Exposure Limit (AEL) of the agent. That means all the following techniques require further testing (such as Mass Spectrometric analysis (see below)) on-site, but especially in an off-site laboratory for their results to be entirely conclusive.

Colorimetric detection

Colorimetric techniques are among the cheapest and easiest ways to detect chemical agents and have been widely used as an immediate response to potential chemical attacks.¹⁵ In order to sample the agent present in an area, manual suction pumps are used to draw air onto detection papers, tickets, or tubes in a method referred to as 'manual vapour detection'.¹⁶

Colorimetric detection is usually sufficient in the case of nerve and blister agents and provides an immediate reading of the substances tested. It can identify specific types of agents (nerve or blister) through a reaction between the molecules of the agent and a solution. Varying chemical properties (such as acidity levels) of targeted agents will produce a distinctive colour (visible to the naked eye or through photometric instruments) when coming into contact with the solution.¹⁷

Detection paper is preferred in the case of liquid agents and is used to reveal immediate threats as well as to allow the mapping of contaminated areas. It contains a dyed solution producing a pigment through a PH indicator (based on acidity).¹⁸ Dyes react to the acidity level, which vary according to the chemical properties of the substance tested. It is used for three chemical warfare agents classes: blister agents (red), G-series nerve agents (yellow), and V-series nerve agents (green/black).¹⁹

One advantage is the rapidity of response: only 30 seconds with some papers. However, detection paper is not always reliable due to its tendency to show

“An array of field detection techniques are available for first responders in situations in which chemical weapons are suspected to have been used.”

17. Fox S. et al (2007), Emergency First Responders' Experience With Colorimetric Detection Methods. Idaho National Laboratory. Available: [http://www.inl.gov/technicalpublications/Documents/3828488.pdf]

18. Ibid.

19. Colours depend on the make of paper/reagent added.

20. Sferopoulos R. (2009), A Review of Chemical Warfare Agent (CWA) Detector Technologies and Commercial-Off-The-Shelf Items. Australian Government Department of Defence. Available: [http://www.dtic.mil/cgi-bin/GetTRDoc?AD=ADA502856]

21. Fatah A. et al (2007), Guide for the Selection of Chemical Detection Equipment for Emergency First Responders, 3rd Edition. U.S. Department of Homeland Security. Available: [http://www.nist.gov/oles/upload/DHS_100-06ChemDetFinReport_3-20-07.pdf]

“Collection may take time, especially in a conflict zone where many people have been affected.”

false positives. It also becomes unreliable on degradation products, which develop as chemicals are absorbed into their external environment.

Ion-Mobilisation Spectrometry (IMS)

IMS technology allows molecules to be analysed according to their mass, mobility and charge.²⁰ It is used for chemical agents in the form of vapour and their degradation products. IMS instruments operate by allowing chemical molecules to be ionized through a method known as Atmospheric Pressure Chemical Ionisation (APCI). This often requires a radioactive source (usually nickel-63 or americium-241).²¹ Ions pass through an electric field with a drift gas, and can be distinguished according to their velocity. The detector is then able to provide information on the chemical agents sampled.

Advantages of IMS instruments include portability and reliability, with a short response-time. Military and civil authorities have generally used them to detect drugs and explosive substances. However, environmental conditions such as high humidity can affect the accuracy of the readings and increase the chance of false positives. IMS instruments also have low selectivity.

Surface Acoustic Wave (SAW)

SAW detectors measure acoustic waves in piezoelectric materials on which a coat sensitive to specific chemicals (or ‘polymeric’ film) has been added.²² As polymeric films each absorb particular chemicals, their mass change. Piezoelectric materials are able to respond to such changes by altering frequency. Such signals provide information on the identity of the chemical agent and its concentration levels. Nerve agents can usually be detected below 1 mg/m³, and the detection limit for blister agents is between 1-2 mg/m³.²³ SAW sensors are relatively low cost and reliable.²⁴ They can detect and identify a range of chemical agents, although polymeric films can be affected by temperature and humidity variations.

Identification

Confirming an alleged use of chemical warfare agents is not simple. Firstly, samples need to be collected. Sources may come from soil, dust, water, weapons fragments, clothing, blood and other bodily fluids and tissues from victims. These samples may contain traces of the chemical warfare agents themselves or the compounds into which the chemicals degrade, or biomedical adducts of the chemical warfare agents. But collection may take time, especially in a conflict zone where many people have been affected. Samples will then need to be sent to special laboratories for rigorous analysis to identify the chemical warfare agents used.²⁵ Furthermore, nerve agents are highly toxic and are lethal even in trace amounts; this coupled with the volatility, the ability of substance to vaporize, of G-agents, makes sample-handling potentially hazardous, and so significant caution, protective equipment and special procedures are required.

Other issues surround the collection of samples from a battlefield. Shelling activity can cause people to evacuate an area, taking their wounded with them, and thus making them unavailable to inspectors visiting the attacked site. This is an issue for sample collection as the bodies of victims provide some of the strongest evidence for the use of chemical warfare agents. For example, sarin, soman, tabun and VX are organo-phosphorus chemical warfare agents, and so their influence on the human body is well known—they strongly inhibit cholinesterases.²⁶

Given the range of physical characteristics—varying degrees of volatility and posing both a vapor hazard as well as a liquid contact hazard—chemical warfare agents have been amenable to analytical techniques commonly employed in most environmental analysis, namely gas chromatography (GC) and liquid chromatography (LC) with a variety of detectors including mass spectrometry (MS).²⁷

Nerve and blister agents undergo hydrolysis in the environment, and methods are required for indication of their degradation products. These degradation compounds are significant because many

22. Ibid.

23. Surface Acoustic Wave (SAW) Sensing /Technology for the Detection of Nerve and Blister Agents, MSA. Available: [http://s7d9.scene7.com/is/content/minesafetyappliances/07-2093_SAWWhite-Paper]

24. Sferopoulos R. (2009), A Review of Chemical Warfare Agent (CWA) Detector Technologies and Commercial-Off-The-Shelf Items. Australian Government Department of Defence. Available: [http://www.dtic.mil/cgi-bin/GetTRDoc?AD=ADA502856]

25. Patricia Lewis, ‘Syria: Chemical Weapons and the Spectre of War’. Available from: <http://www.chathamhouse.org/media/comment/view/194007>

26. Y. Seto, 2002. ‘Forensic Investigation of the Sarin Gas Attacks in Japan’, in: Rodrigo Yepes-Enriquez and L. Tabassi (ed.), Treaty Enforcement and Inter-

of them would not normally be found in environmental samples and their identification strongly suggests prior presence of chemical warfare agents. However, ultimate proof would be identification of the chemical warfare agents themselves. The degradation products of chemical warfare agents, in particular of nerve agents, are non-volatile hydrolysis products that must be derivatised in preparation for GC analysis.²⁸

Chromatography

Gas chromatography (GC) is an analytical technique that is used to separate volatile organic compounds from each other. A gas chromatograph has a separation column containing a stationary phase inside an oven, a detector, and a data recording system and is using a gas-like helium or nitrogen as a mobile phase.

Contaminated samples containing chemical warfare agents typically contain multiple components that are best characterised following chromatographic separation. These samples usually fall into one of the following general categories:

- Munitions or munitions fragments (e.g., neat liquid or artillery shell casing);
- Environmental (e.g., soil, water, vegetation or air samples);
- Man-made materials (e.g. painted surfaces or rubber); and,
- Biological media (e.g., blood or urine).

The speed and accuracy of the analysis depends on the amount of sample preparation required to obtain a suitable sample or extract for the chromatograph. For example, where neat liquid has been collected, the sample normally only requires dilution with a suitable solvent prior to analysis. Other samples, such as environmental samples, require solvent extraction and concentration before an analysis can be performed or even a more elaborate sample preparation.²⁹

Mass spectrometry

Mass spectrometry (MS) is an analytical technique that measures the masses of

molecular fragments. It is used for determining the masses of molecules and their fragments and gives a 'fingerprint' of a chemical.³⁰ MS is useful for the detection and characterisation of chemical warfare agents, their precursors (chemicals used to manufacture them), their degradation products and related compounds.³¹

This is a tried and tested method of analysis. Registered mass spectra of emerging peaks from a GC-MS are routinely compared to libraries of mass spectra available from the OPCW, commercial standard libraries and defence community libraries.³²

Methods have been developed such as chemical ionisation (CI) as a complementary ionisation technique that has generally yielded molecular ion information for chemical warfare agents and has also been used to identify related compounds and impurities in the agent munitions and environmental samples.³³ The characterization of these compounds, once coupled with other analytical techniques, is an important tool for OPCW analysis since the data may provide an indication of the origin of the sample, the method of manufacture or the degree of sample degradation.

Conclusion

This month, August 2013, appears to have witnessed a chemical attack on a scale not seen since the Iraqi air force killed 6,000 with chemical weapons in the Kurdish town of Halabja, 30 years ago. As the UN inspection team prepares to report on its findings from Ghouta, this briefing paper is being released with the intention of providing a factual overview of the methods used in chemical weapons detection and identification both in the early and later stages after an alleged attack. It is hoped that this will help readers to understand the challenges posed to the inspectors currently inside Syria, while also assisting readers in interpreting the array of reports and commentaries currently emerging with respect to the recent and ongoing events there.

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national Cooperation in Criminal Matters: with Special Reference to the Chemical Weapons Convention (T.M.C. Asser Press: The Hague), p. 302

27. PA. D'Agostino and C.L. Chenier, 2006, p. 5

28. Ibid.

29. Ibid.

30. <http://www.chem.pitt.edu/facilities/mass-spec-trometry/introduction>

31. PA. D'Agostino and C.L. Chenier, 2006, p. 8

32. Ibid.

33. Ibid.

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About this paper

In this brief, David Cliff, Russell Moul and Ariane Jugieux consider the legal and technical aspects of the detection of chemical weapons use—both in the immediate and later stages after an alleged attack. The brief looks at the UN Secretary-General's 'Mechanism for Investigation of Alleged Use of Chemical and Biological Weapons' (as being put into practice in Syria at the time of publication) and also at the various technical tools available to investigators and first-responders.

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