



## Study of the deposition of ammonium perchlorate following the static firing of MK-58 rocket motors

Sonia Thiboutot, Guy Ampleman, Marie-Claude Lapointe, Sylvie Brochu, Marc Brassard, Robert Stowe, Rocco Farinaccio, Annie Gagnon, André Marois and Thérèse Gamache, DRDC Valcartier

#### Defence R&D Canada - Valcartier

Technical Report DRDC Valcartier TR 2008-240 October 2008



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#### **Defence R&D Canada – Valcartier**

Technical Report
DRDC Valcartier TR 2008-240
October 2008

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#### **Abstract**

Static firings of AIM-7 rocket motors were conducted in June 2006 at the DRDC Valcartier test site to measure the environmental dispersion of ammonium perchlorate (AP) during normal burning conditions. The recent stringent threshold criterion for perchlorate in drinking water dictates that the sources of this contaminant be better defined. The AIM-7 Sparrow missile, which is propelled by an MK-58 AP-based motor, has been the main medium range air-to-air missile used by both the US and Canadian fighter jets for many years. Fifteen MK-58 motors were obtained for this study from the Canadian inventory and statically fired on a test bench to measure the residues that were expelled during combustion. The field set-up to catch perchlorate particles was based on a combination of aluminium witness plates and aqueous traps located in the exhaust plume area. The tests were conducted over four days and sampling was achieved after the burning of two, three, four and six motors. The set-up allowed the collection of a large portion of the exhaust plume, and perchlorate was detected in most of the samples collected, due to the use of a very sensitive analytical method. The results obtained were consistent and reproducible, and it was estimated that only 2 mg of perchlorate was deposited per MK-58 motor. Considering the motors flight range, it is concluded that their use in live fire training does not contribute to the accumulation of perchlorate in the environment.

#### Résumé

Des mises à feu statiques de moteurs-fusées AIM-7 ont été réalisées en juin 2006 sur le site d'essais de RDDC Valcartier, pour mesurer la dispersion environnementale du perchlorate d'ammonium (PA) dans des conditions normales de brûlage. Le critère de seuil rigoureux émis récemment concernant le perchlorate dans l'eau potable implique de déterminer les sources potentielles de ce contaminant. Le missile Sparrow AIM-7, propulsé par un moteur MK-58 à base de PA, est le principal missile air-air de moyenne portée utilisé sur les avions de chasse américains et canadiens depuis de nombreuses années. Quinze moteurs MK-58 provenant de l'inventaire Canadien ont été lancés de façon statique avec un banc d'essai fixe pour mesurer les résidus déposés durant la combustion. Les essais ont été réalisés sur une durée de quatre jours et l'échantillonnage a été effectué après la mise à feu statique de deux, trois, quatre et six moteurs MK-58. La stratégie de collection des particules a été élaborée en combinant des plaques témoins en aluminium et des trappes d'eau dispersées dans la zone du panache de fumée. Cela a permis la récolte d'une large portion du panache de dépôt et le perchlorate a été détecté dans la plupart des échantillons compte tenu d'une méthode d'analyse très sensible. Les résultats obtenus sont cohérents et reproductibles et il a été estimé que chaque moteur MK-58 dépose seulement 2 mg de perchlorate. Considérant la distance de vol de ces moteurs, on peut conclure que l'utilisation de ceux-ci dans l'entraînement en tir réel ne contribue pas de façon significative à l'accumulation de perchlorate dans l'environnement.

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#### **Executive summary**

# Study of the deposition of ammonium perchlorate following the static firing of MK-58 rocket motors:

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**Introduction or background:** Live-fire training is an essential activity to maintain the readiness of our troops. R&D has been dedicated in the past years to the characterization of various types of training ranges and to the measurement of the accumulation of munitions-related residues in the environment. Training ranges have been visited and protocols have been drafted to efficiently characterize them. The munitions-related residues that have been studied extensively up to now are explosives, gun propellants, anti-tank rocket propellants, and heavy metals. Perchlorate-based energetic materials have not been scrutinized yet for their potential accumulation in the environment, and this needed to be addressed. Ammonium perchlorate (AP) is an oxidizer widely used in solid rocket propulsion, and recently it has been detected in various groundwater reservoirs in the United States. In Canada, Environment Canada is currently putting together a database specifically on the levels of perchlorate in Canadian groundwater, and we have detected it in a few groundwater wells at a few training areas. It is a newly identified and important contaminant of concern, due to its potentially adverse human health impacts and its very low threshold criteria. Therefore, it was imperative to verify if the live-fire training with AP-based rockets could lead to the accumulation of perchlorate in the environment. The goal of the present study was to conduct the static firing of 15 MK-58 rocket motors and to measure the amount of perchlorate that was deposited. This rocket motor is used on the AIM-7 missile, which is widely used by both Canada and the United States as a medium range air-to-air weapon on their respective fighter jets. The study was held in June 2006 at the DRDC Valcartier test site.

**Results:** A test set-up was designed to collect perchlorate particles ejected during the static firings on a bench to concentrate the motor plumes in the smallest possible area. The field set up was successful and perchlorate particles were effectively caught in the particle traps. Our set-up indicated that each motor deposited 2.4 mg of perchlorate in the environment per motor. Considering that each MK-58 motor contains 47 kg of AP, this means that we have measured a dispersion of 2.4 mg out of 47,000,000 mg, resulting in 4.25 x  $10^{-6}$  % of perchlorate deposited per motor.

**Significance:** It was demonstrated that, during normal burning conditions, perchlorate-based solid propellant motors generate very small quantities of perchlorate particles in the environment. If we consider the normal range of these rockets, reported to be on the order of 50 km and the levels of perchlorate generated, the environmental impact of live-fire training with perchlorate-based motors is negligible in terms of the dispersion of perchlorate particles.

**Future plans:** No other perchlorate deposition trials are planned, considering the high burning efficiency measured.

#### **Sommaire**

# Study of the deposition of ammonium perchlorate following the static firing of MK-58 rocket motors:

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Introduction ou contexte: L'entraînement en tir réel est une activité essentielle pour maintenir la vigilance de nos troupes. La R et D a été consacrée dans les années passées à la caractérisation de différents types de sites d'entraînement et des résidus de munitions accumulés dans l'environnement. Plusieurs sites d'entraînement ont été visités et des protocoles ont été rédigés pour leur caractérisation efficace. Les résidus qui ont été étudiés jusqu'à maintenant sont les explosifs, poudres à canon, propergols pour moteurs-fusées antichars et métaux lourds. Les propergols à base de perchlorate n'ont pas été examinés à ce jour en ce qui concerne l'accumulation de résidus dans l'environnement, et cette évaluation devait être effectuée. Le perchlorate d'ammonium (PA) est un oxydant largement utilisé dans la propulsion des moteursfusées solides, et récemment il a été détecté dans différents réservoirs d'eaux souterraines aux États-Unis. Environnement Canada établit une base de données visant à déterminer le niveau de perchlorate dans les eaux souterraines au Canada et notre équipe en a détecté dans quelques puits d'eaux souterraines sur des sites d'entraînement. Il est important de tenir compte de ce contaminant nouvellement identifié étant donné l'impact négatif potentiel sur la santé humaine et le critère de seuil rigoureux. Il était impératif de vérifier si l'entraînement en tir réel avec des fusées à base de PA permet l'accumulation du perchlorate dans l'environnement. Des tirs statiques de moteurs-fusées MK-58 ont été effectués ainsi que des mesures de la quantité de perchlorate relâché. Ce moteur-fusée propulse le missile AIM-7, qui est largement utilisé au Canada et aux Etats-Unis comme arme aérienne de moyenne portée sur les avions de chasse. L'étude s'est déroulée en juin 2006 sur le site d'essai de RDDC Valcartier.

**Résultats:** L'essai a été élaboré afin de récolter les particules de perchlorate éjectées durant la mise à feu statique de moteurs à partir d'un banc d'essai fixe. Cela a restreint le panache de projection à la plus petite superficie possible. Le protocole utilisé a permis la capture efficace des particules projetées dans les trappes. Nous avons démontré que chaque moteur libère 2,4 mg de perchlorate dans l'environnement. Si on considère que chaque moteur-fusée contient au départ 47 kg de perchlorate, sa combustion génère 4,25 x 10 <sup>-6</sup> % p/p de perchlorate.

**Importance:** On a démontré que, dans des conditions d'utilisation normale de ces moteurs, la quantité générée de perchlorate dans l'environnement est très faible. Si on considère la distance de vol normale de ces moteurs-fusées, évaluée à environ 50 km, et le niveau de perchlorate généré, l'impact environnemental de l'entraînement en tir réel avec un moteur à base de perchlorate est négligeable quant à la dispersion de particules.

**Perspectives:** Aucun autre essai de mesure du dépôt de perchlorate n'est planifié compte tenu de l'efficacité de combustion mesurée.

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#### 1 Introduction

Modern solid composite rocket propellants are mostly based on an ammonium perchlorate (AP) oxidizer dispersed in either hydroxyl- or carboxy-terminated polybutadiene (HTPB or CTPB). AP is a very good oxidizer which leads to high-performance, high burning rate rocket motors, and as such, it has been used extensively in the past in many solid rocket propellant formulations, including its use by NASA Space Shuttle (Ref.1). Perchlorates are also used in a number of other applications including as a component of fireworks, pyrotechnics, flares and explosives. Other uses include pharmaceuticals for hyperthyroidism, gas generators, electrolytes for lithium cells, and as chemical reagents. The occurrence of perchlorate in the environment is principally anthropogenic in nature. There are major drawbacks to the use of AP as an oxidizer in rocket propellants. The combustion of AP generates hydrochloric acid, which is a highly corrosive and non-environmental by-product that might generate acid rain, toxicity, and decrease the air quality (Ref. 2).

This gaseous acidic emission is also easy to track, since it may lead to visible white secondary smoke in the missile plume. Efforts have been dedicated in the recent years to replace AP by other oxidizers that would avoid the production of hydrochloric acid, such as ammonium nitrate (Refs. 3, 4). Unfortunately, the burning rates of ammonium nitrate-based propellants were insufficient and, despite large efforts several years ago, did not lead to acceptable rocket propellant formulations. More recently, ammonium dinitramide (ADN, NH<sub>4</sub> N(NO<sub>2</sub>)<sub>2</sub>), was introduced as an oxidizer in order to produce a new generation of environmentally friendly rocket propellants and for spacecraft propulsion (Refs. 5, 6). SERDP presently sponsors research in the area of the synthesis, evaluation, and formulation of oxidizers as alternatives to AP in missile propulsion applications (Ref. 7A).

Another strong drawback of the use of AP as an oxidizer in rockets is its potential dispersion in the environment upon its production, use or demilitarization at the end of the rocket service life. Perchlorate salts are water soluble and not significantly adsorbed by soil minerals or humic substances and hence are highly mobile in the environment. This chemical represents a health concern because when ingested, it can disrupt the thyroid function by competitively inhibiting iodide transport (Ref. 8). Due to its potential adverse health effect, the US Environmental Protection Agency (EPA) included perchlorate on the Drinking Water Candidate List 2 (Ref. 9). Health and ecological risk associated with perchlorate exposure are presently under investigations and SERDP is sponsoring projects on the study of the ecological risks of AP (Ref 7B). Studies are also conducted on potential treatments for the remediation of perchlorate in the environment (Refs. 10, 11). The debate on the acceptable levels of perchlorate in drinking water led to the adoption of a more stringent threshold criterion for perchlorate in drinking water. The discovery of widespread low levels of perchlorate in various aquifers in the US (Ref. 10) has engaged the governmental regulatory agencies in an intensive analysis regarding perchlorate toxicology and the recommended daily dose exposure for humans (Refs. 12, 13). A reference dose of 0.0007 mg/kg/day of perchlorate was adopted by the US EPA in February 2005, which translated into a drinking water equivalent level of 24.5 µg/kg (Ref. 14).

These findings in turn dictate that the sources of this contaminant in the environment must be better defined in order to minimize its further dispersion. There are very few papers in the literature on the dispersion of perchlorate particles from the burning of AP-based rocket motors.

Only one study was published, on the development of a method to predict potential perchlorate releases from accidental launch failures (Ref. 15). However, previous studies conducted on the dispersion of gun propellant residues in the environment following the firing of artillery and mortar rounds demonstrated that measurable amounts of gun propellant residues can be found near the gun exit (Refs. 16-17).

The goal of this trial was to determine if the firing of rocket motors in their normal firing conditions can lead to the dispersion and accumulation of unburned AP in the environment. To achieve this, the Canadian inventory was reviewed to identify a representative AP-based rocket motor that is also used by the American military. AIM-7 missile rocket motors were identified as such, and a request was made to the Director General of Aerospace and Engineering Program Management (DGAEPM) to acquire 15 motors to conduct a representative static burn tests. This request was accepted, and the 15 motors were shipped to Defence Research and Development Canada (DRDC) Valcartier from Cold Lake Air Force Base.

The AIM-7 Sparrow missile is a radar guided air-to-air missile with a high explosive warhead (Fig. 1). It is a missile widely used by Canadian, American and North Atlantic Treaty Organization (NATO) forces and it has been the main supersonic medium range air-to-air missile of U.S. and Canadian fighter jets for many years (Ref. 18). It is propelled by an AP/CTPB solid propellant dual thrust rocket motor (Ref. 19). The Canadian version of the AIM-7 rocket is propelled by MK-58 Mod 5 motors.



Figure 1: AIM-7 Rocket fired from an F18 fighter jet.

Prior to the static firing of the AP-based MK-58 motors, our set-up was tested with Air Defence Anti-Tank System (ADATS) motors firing for tests that were conducted by the Propulsion Group to study their low temperature operation (Ref. 20).

This paper describes the static firing of the MK-58 Mod 5 rocket motors on a test bench at the DRDC Valcartier test area and the measurement of the AP residues expelled during combustion. Static firings of the rocket motors were conducted between June 20<sup>th</sup> and June 28<sup>th</sup> 2006, and were co-sponsored by SERDP, DGAEPM, and the Sustain trust 12 SG.

#### 2 Experimental

#### 2.1 Field Work

The trial was conducted at DRDC Valcartier Test Range, located within the Garrison Valcartier. ADATS motor firing was held between June 14<sup>th</sup> and 16<sup>th</sup> 2006, while MK-58 tests were conducted between June 20<sup>th</sup> and June 28<sup>th</sup>. The ADATS trial was conducted by the Propulsion Group to test the low temperature (-40°C) operation of six ADATS rocket motors from various production lots. We used these firings to validate and prove our field set-up, in preparation for the MK-58 trial. If feasible, sampling of the RDX-based ADATS residues would have been conducted, but it was not achieved due to various technical problems encountered. The MK-58 trial was conducted in four days, on June 20<sup>th</sup>, 22<sup>nd</sup>, 27<sup>th</sup> and 28<sup>th</sup> where two, three, four and six motors were fired, respectively. The meteorological conditions prevailing on these days were difficult and are reported in Table 1. No firing was conducted on June 21<sup>st</sup> due to heavy rain conditions.

#### 2.2 Material

Six ADATS motors (Fig. 2) were statically fired after a conditioning period of 48 hours at -40°C. Fifteen MK-58 Mod 5 (Lot QZC) rocket motors (P/N 1131AS200-1, NSN 1337-01-150-7939) were obtained from the Director General Aerospace Engineering Program Management (Fig. 3). They were allocated to DRDC Valcartier's environmental study and sent from 4 Wing-Cold Lake depot, approximately one month prior to the trial. Aluminium witness plates, 1 m x 1 m x 3 mm, were placed in front of the rocket nozzle to collect potential residues (Fig. 4). They were wiped with cotton gauze, using distilled water to wet them when dry meteorological conditions prevailed. Water traps were also used in the second set-up to catch and trap perchlorate particles. Commercially available rectangular aluminum pans of 39.2 x 32.3 x 7.9 cm, were buried in the soil at a depth until flush with the surface and filled with distilled water (300-500 ml). Rocks were placed in the pans to stabilize them and prevent their movement by the rocket motor plume (Fig. 5). The water in the traps was collected using 1-1 amber glass containers and the exact volumes were measured. On rainy days, the rain water accumulated in the traps and the total volume of water was collected. On June 20<sup>th</sup>, 16 traps were used, while 20 traps were used per day afterward.



Figure 2: ADATS Motor.



Figure 3: MK-58 motor.



Figure 4: Aluminium witness plate.



Figure 5: Particle trap filled with water.

#### 2.3 Propellant composition

#### 2.3.1 ADATS motors

ADATS motors use cross-linked propellant mixed with RDX crystals. They contain 19.6 kg of cross-linked double based propellants, with 7.8 % by weight of binder, 62 % of RDX, 25.88 % of plasticizers, and a few percent of thermal stabilizers, ballistic modifiers and acoustic additives (Ref. 20).

#### 2.3.2 MK-58 motors

The AIM-7 MK-58 rocket motor is 203 mm in diameter by 1514 mm long. The case is constructed from steel, has a wall thickness of 1.880 mm and contains 61 kg of CTPB-based propellant. It uses a dual grain propulsion system with two types of propellants to provide a boost-sustain thrust profile. Both propellants are based on AP dispersed in a CTPB matrix, but their formulations are slightly different in terms of relative percentage of ingredients. The ratio of sustain to boost propellant is 70 to 30 percent by volume (Ref. 21). Other ingredients include plasticizers and phenyl-beta-naphtylamine (PBNA) as an anti-oxidant. This latter compound is present at less than 1% by weight in the propellant but it might be considered as a health hazard since it has adverse toxicological impacts. It was not included as an analyte in the present study, but depending on the levels of residues deposited, it could be important in further studies. The detailed formulations of these propellants are considered proprietary information. However, the fact that MK-58 motors contain 47 kg of AP can be disclosed.

#### 2.4 Field set-up and sampling strategy

The initial set-up was tested by firing the six ADATS motors before firing the MK-58 motors. This first set-up was based on a preliminary study conducted in 2004 that evaluated the gaseous emissions form the static firing of ADATS motors (Ref. 22). The deposition area was secured by using concrete blocks in a "U shape" (11 blocks of 2' by 2' by 6'), installed 8 m away from the motor nozzle (Fig. 6). These concrete blocks were intended to protect the witness plates against the blast from the motors and to reduce the disturbances at the surface of the soil. A concrete pad 17 m wide by 12 m long was located directly in front of the test stand and five aluminum witness plates were screwed to its surface, 10 m behind the motor nozzle and 1.5 m from each other. Three polyethylene tarps were used as soil cover to minimize the potential cross contamination from the underlying soil and were installed from the concrete pad to 35 m from the nozzle. Twenty witness plates were equally spaced and placed on the three tarps at respectively 13, 16 19, 22 and 25 m from the nozzle (Fig. 7). Sandbags were deposited on the leading edge of the aluminum witness plates to stabilize them during the firing of the rockets (Fig. 8). The tarps were also tied up with sandbags. Prior to both ADATS and MK-58 static firing, the fire department of Garrison Valcartier was tasked to wash the entire concrete surface and concrete block to minimize potential cross contamination from past trials (Fig. 9). They washed the surface on June 14<sup>th</sup> and June 20<sup>th</sup>.



Figure 6: Installation of the concrete wall.

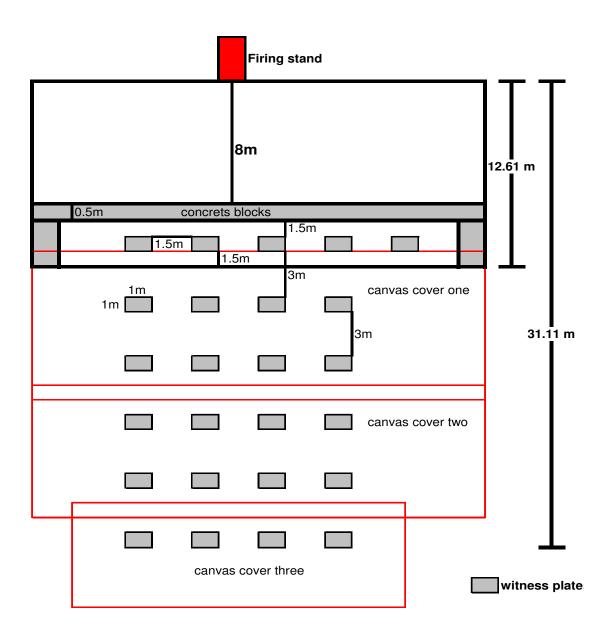


Figure 7: Initial set- up diagram.



Figure 8: Initial test layout.



Figure 9: Washing of the concrete surfaces.

The initial set-up failed in allowing the collection of residues from the static firings. Furthermore, as a result of the heat developed by the rocket blast, seven sandbags and the first tarp melted (Fig. 10). Moreover, visual observation of the firings proved that our plates were not correctly located, since the plume deposition area was a lot larger than expected (Fig. 11). The ADATS rocket blast was obviously more powerful than expected, and a realistic idea of how far and fast the blast of an MK-58 motor would carry was required. A simulation of plume velocity versus distance was carried out to show that at a distance of 200 meters, the speed of the blast would still be 10 m/s. Consequently, no residues were sampled from the ADATS firings because the plates were too close and the tarp melted on many of them. The use of witness plates as the receptacle for particulate material in such a highly turbulent environment was also questioned.



Figure 10: Initial layout after ADATS static firings.



Figure 11: ADATS static firing.

Keeping this information in mind, it was decided to extend the sampling template up to 200 meters from the firing stand and to remove the polyethylene tarps. Witness plates were placed in a triangular pattern using a wider sampling area. The plates were installed at 10, 50, 100 and 150 m from the motor nozzle (P0, P1, P2 and P3 rows). Water traps were also used as receptacles for perchlorate particles and spread out evenly in the plume deposition area (W1 to 20). These traps were used to minimize the potential of losing the particles due either to rain conditions, wind or turbulence. The second field set-up, which included 16 water traps, is illustrated in Figure 12 was used on June 20<sup>th</sup>. A third and final set-up was then used until the end of the trial by adding an additional four water traps (Figs. 13, 14). Our set-up was correctly positioned based on the visual observation of the plumes generated by the MK-58 firings (Fig. 15).

Wipes from witness plates in the same rows were combined, based on the small area covered by each plate (1  $\text{m}^2$ ). They were designated as P0 to P4-ALL-AIM06-trial. As such, the area sampled per row represented a total area of 5, 4, 7 and 9  $\text{m}^2$  respectively for rows labelled P0, P1, P2 and P3. Water trap samples were labelled W-1 to 20-AIM06- trial #. The area covered by one water trap was 0.127  $\text{m}^2$ .

Finally, composite soil samples were also collected in front of the same area, using more than 100 sub-samples in three areas of 20 m by 20 m in front of the firing line (A, B and C). Three field replicates were collected before the test and three at completion of the four days.

On a daily basis, the following was accomplished:

#### Day one, June 20th, test # 1.

Two motors were fired at 14h00 and 14h20. The firings were done under light rain and then heavy rain prevailed. Witness plates were not sampled due to heavy rain and flooding of the plates. The 16 water traps were sampled after the two motors were fired. The labelling was W 1 to 16, AIM06-1.

#### Day two, June 22<sup>nd</sup>, tests # 2, 3 and 4.

Three motors were fired at 9h20, 9h55 and 10h35 under light rain conditions. Witness plates were sampled after each firing. The labelling was: P0 to P3-AIM06-ALL-2 to 4. Nineteen water traps were sampled (trap # 20 was blown away by the blast) after the three firings and were labelled W 1 to 19-AIM06-2.

#### Day three, June 27th, tests # 5 and 6.

Four motors were fired at 10h00, 10h50, 11h17 and 11h45 under light rain conditions. Witness plates were sampled after the first firing (test #5) and then after the three remaining motors (test #6). They were labelled P0 to P3-AIM06-5 and 6. Twenty water traps were sampled after the four motors and were labelled W 1 to 20-AIM06-3.

#### Day 4, June 28<sup>th</sup>, test # 7, 8, 9 and 10.

Six motors were fired under cloudy conditions at 8h46, 9h22, 9h50, 10h16, 10h51 and 11h20. Witness plates were sampled after the first motor (test # 7), the second motor (test # 8), the third and fourth motors (test #9) and the fifth and sixth motors (test #10). They were labelled P0 to P3-AIM06-7, 8, 9 and 10. Twenty water traps were sampled after the six motors and were labelled W 1 to 20-AIM06-4.

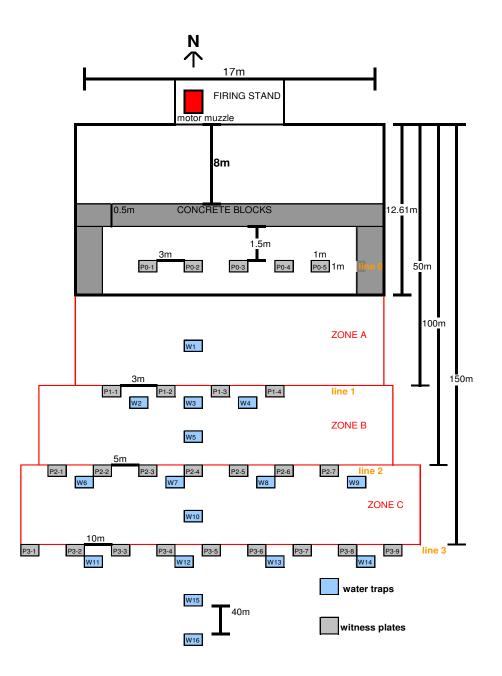


Figure 12: Second set-up diagram.

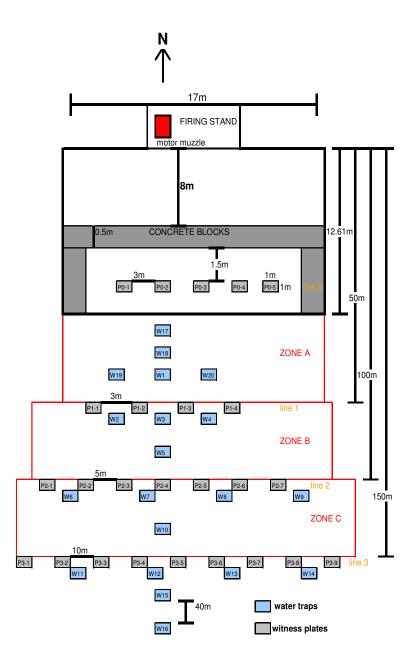


Figure 13: Third set-up diagram.



Figure 14: Third set- up.



Figure 15: MK-58 static firing.

#### 2.5 Test stand

A mobile test stand, designed to be installed rapidly on an open-air experimental site, was used during the firing of six ADATS and 15 AIM-7 rocket motors (Figs. 16, 17). This installation was designed and built by DRDC to study the plumes of different types of rocket motors. A special mounting system was fabricated to safely retain the MK-58 motors during firing (Fig. 18). This installation allows the user to measure various characteristics of rocket motors such as thrust and chamber pressure while minimizing masking of the exhaust plume by the test stand hardware from any aspect angle. The height of this test stand can be varied from 1.0 to 3.3 meters from the ground. The assembly can also rotate about a vertical axis if required, and there is a provision to point the motor upwards at a 45-degree angle. This test stand is equipped with tie-rods to stabilize the test stand laterally, and rocket motors of up to 90 kN of thrust may be tested.



Figure 16: Test stand.



Figure 17: View from the rear of the test stand.



Figure 18: MK-58 motor collar.

For ADATS and MK-58 firings, the motors were positioned horizontally at 1.5 m off the ground. Unobstructed views of the motor were possible from at least 200 m in any direction and allowed various monitoring and high speed recordings of the firings (Fig. 19).



Figure 19: Test stand at a distance.

#### 2.6 Motor ignition

All motors were ignited remotely and supervised by an experienced ammunition technician. All personnel were either in special shelters or at the trailer, located approximately 300 m from the test bench. The ignition system consists of three batteries mounted in parallel placed at the base stand and connected to a 120-volt power supply. When the power supply was connected to the AC system and engaged by the ammunition technician situated in the trailer, the loop closed and ignited the squib of the rocket (Fig. 20). A five-minute period had to be respected for safety after each motor firing prior to inspection by the ammunition technician who then gave the authorization to come back to the test site for sampling. A delay of 5 to 10 minutes resulted between firing and sampling.

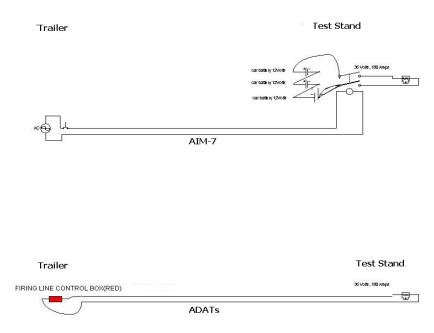


Figure 20: Ignition system.

#### 2.7 High speed camera

All motor firings were recorded using a monochrome high speed camera from Canadian Photonics Labs. It has a maximum resolution of 504 X 504 pixels with a camera buffer memory of 8.6 gigabytes and a maximum frame rate speed of 91,250 frames per second (fps) (resolution of 504 X 24). The camera's data acquisition and control system software was supplied with portable PC hardware. The communication between the portable PC hardware and the camera was achieved with 100-gigabit Ethernet connectivity. An external trigger source was used to manually trigger the camera during the event. The camera was located 70 m away, to the side and slightly behind the thrust stand, at 120 degrees for the ADATS and at 90 degrees for the MK-58. The main focus of the use of this camera by the Propulsion Group was to evaluate failure, if any, at the blast tube area of the rocket motor in the ADATS firings. The team also recorded the MK-58 firings to see any evidence of perchlorate particle ejection. The camera was set to operate at 1000 fps at maximum resolution. However, due to the greater focal length of the lens and consequently less light being available, the shutter speed and gain were increased to 700 and 600 µs, respectively. The aperture was set at F8.0 for maximum opening of the lens. The recording time of each event was set between 5-7 seconds for ADATS and approximately 20 s for the MK-58 by manual trigger. The field of view for the MK-58 was cropped to 504 x 120 pixels, which allowed a more manageable camera memory buffer for the 20-s recording.

#### 2.8 Sample extraction and analysis

Water collected in water traps was brought to DRDC Valcartier laboratory at the end of each day. Samples were immediately filtered using disposable 60-ml plastic syringes and a 0.45-µm syringe filter, and their exact volumes were measured and are reported in Table 2. An aliquot of 20 ml was poured into 50-ml high density polyethylene containers. Samples were stored with a large headspace to avoid anaerobic degradation, kept at 4°C and sent within 48 hours by express transport to Environment Canada for analysis. One field blank was included by processing a water sample in the exact same way as for the test samples and a laboratory blank was included by processing distilled water in the laboratory with the same procedure used for the water trap samples. They were labelled respectively P-5-AIM06-B2 and P-5-AIM06-B1.

The witness plate wipes were brought to the laboratory at the end of each day. All samples were wet due to the rainy conditions prevailing on the first three days of trial. A volume of 100 ml of distilled water was added to the jars, which were vigorously agitated manually three to five times and then sonicated for 30 minutes. The water extracts and the wipes were filtered by putting the soaked wipes into 250-ml plastic syringes using 0.45-µm syringe filters. The wipes were further rinsed by adding 50 ml of distilled water to collect any residual perchlorate. The total water volumes extracted were noted and are reported in Table 3. An aliquot of 20 ml was poured into a 50-ml high density polyethylene container. Samples were stored with a large headspace to avoid anaerobic degradation, kept at 4°C and sent within 48 hours by express carriage to Environment Canada for analysis. Soil samples (200 g) were extracted using distilled water by sonication at room temperature for one hour, the water was filtrated and the extracts analyzed.

The analysis was conducted by Environment Canada using ion chromatography coupled to a tandem mass spectrometer. Separation was performed using a Dionex 2500 system equipped with an autosampler, a GP50 pump, an EG50 eluent generator and ED50 conductivity detector on a Dionex IONPAC® AS20 analytical column (2 x 250 mm) with a Dionex IONPAC® AG20 guard column (2 x 50 mm). Using an EG50 eluent generator a 35-mM potassium hydroxide solution isocratic eluent was run at a flow rate of 0.35 mL/min. An Agilent 1100 pump was used to tee in 90/10 (acetonitrile/water) at 0.2 ml/min at the mass spectrometer source. Total flow into the mass spectrometer was 0.55 ml/min. Injection volume was 100 µL. Under these conditions, the retention time of perchlorate was ~13.5 min. An API 2000 (MDS Sciex, Ontario, Canada) triple quadrupole tandem mass spectrometer, operated in the electrospray ionization mode, was used for the detection of perchlorate using Analyst® version 1.4 software. The instrument was run in the negative ion mode, and each quadrupole was set to unit mass resolution. Three multiple reaction monitoring transitions were monitored: m/z 99-83, m/z 101-85 (for native perchlorate), and m/z 107-89 (for enriched perchlorate). Quantitation was accomplished using an internal standard method (Cl18O4 -) at 1 µg/L. Instrument calibration was performed by analyzing standards at 0.05, 0.1, 0.2, 0.5, 1, 5, 10, 20 and 40 µg/L in reagent water. To demonstrate that the instrument was properly calibrated throughout the analysis, a continuing calibration verification standard was analyzed every 10 samples. The minimum detection limit achieved was 0.011 µg/L while the practical quantitation limit was 0.05 µg/L.

#### 3 Results and Discussion

#### 3.1 Test Set-up and sampling strategy

The static firing of all motors proved to be successful as all motors were ignited and burned efficiently. The motors were weighted before and after firing, and gave an average mass difference of 61.04 kg. No traces of propellant remained in the motors after ignition, and 100% of the propellant was used.

The collection of perchlorate particles from the static burning of large rocket motors was not an easy task. The first set-up used for the ADATS motors proved to be inefficient since it used a plastic tarp that melted. Moreover, it was clear that our sampling area was not large enough and that most of the plume fell outside of the boundary of our setting. The use of witness plates when compared to the use of water traps showed many weaknesses. In heavy rain or wind conditions, witness plates cannot be used since particles are either washed or blown away. In light rain and soft wind conditions, such as the ones that prevailed the first three days of sampling, their use was still acceptable. The use of witness plates also involves an extraction step, when wipes are extracted by distilled water. In comparison, the use of water traps greatly reduces the risk of analyte loss, as no extraction step is needed. Soil samples were also collected before the test in four large areas within our sampling template, and we planned to sample the same areas at the end of each day. For the first three days, this was not done due to heavy rain. On the fourth day, soil samples were collected and analyzed. Soil extractions may lead to potential interference from other compounds, loss of analyte in the extraction step and cross-contamination from past activity on the test site, so water traps are still considered the best sampling approach.

The global area considered in the final set-up is 4000 m<sup>2</sup>, with dimensions 20 m by 200 m. It was not possible to expand our sampling template area because we reached the limit of the open area with the presence of a concrete wall at one end, and a dense vegetated area at a distance of 220 m from the motor nozzle.

#### 3.2 Meteorological conditions

The meteorological conditions pertaining while the trial was conducted are presented in Table 1. Days one, two and three were rainy, while the fourth day was cloudy without rain. The wind direction and speed are reported, and it has to be pointed out that the highest speed (5 knots) still represents a light wind. It was evident that the use of witness plates was less useful than the water traps for this application, but it was decided to pursue both methods to compare them. On day one, after the firing of the second motor, heavy rain and lightning prevented the collection of samples from the witness plates. Only the water traps were collected. The final volume of each trap was increased due to the large contribution of the heavy rainfall. The light rain conditions prevailing the first three days of testing had the advantage of avoiding dust cross-contamination from the soil, and it helped in wetting the witness plates to improve the adhesion of AP particles.

*Table 1: Meteorological conditions.* 

Date	time	meteorological condition	wind speed/Knots	wind direction	temperature/C
June 20th	14h00	light rain	4	NE	20.8
June 20th	14h20	light rain/heavy rain	4	NE	20.8
June 22nd	09h20	light rain/heavy rain	4	S	16
June 22nd	09h55	rain	3	SO	16
June 22nd	10h35	rain	3	S	16
June 28th	10h00	light rain	2	SO	17.5
June 28th	10h50	light rain	1	SO	17.5
June 28th	11h17	light rain	2	SO	18
June 28th	11h45	light rain	2	SO	18.3
June 29th	8h46	clouds/no rain	4 to 5	N	21
June 29th	9h22	clouds/no rain	5 to 8	NO	21
June 29th	9h50	clouds/no rain	5 to 8	NO	21
June 29th	10h16	clouds/no rain	5 to 8	NO	21
June 29th	10h51	clouds/no rain	5 to 8	NO	21
June 29th	11h20	clouds/no rain	5 to 8	NO	21

We have carefully examined the results in relationship with the wind directions and speed, and no tendency could be established between deposition patterns and wind conditions. As stated earlier, the wind speeds prevailing in the four days of trial were light, and their influence can be considered negligible when compared to velocities in most of the rocket plume.

#### 3.3 Water trap results

The concentration measured in trapped water samples, the volume of water collected, the mass per trap, per square meter and per motor are presented in Tables 2 to 4. Table 2 presents the results for days 1 and 2, while Tables 3 and 4 present the results for days 3 and 4, respectively. The surface area covered by the water in one trap was 0.127 m<sup>2</sup>. Some concentration estimates lie between the minimum detection and practical quantitation limits of the method, so we can conclude that our test set-up allowed the detection of perchlorate, with, in some cases, a lower level of confidence since results are at the limits of the analytical method. If such a high sensitivity method were not used, non-detectable concentrations of perchlorate would have been obtained for most of the samples. Field and trip blanks were included for water traps and the results came back non-detected. In day 1 (Table 2), the concentrations measured were quite homogenous, with the exception of trap number 15, located 172 m from the motor nozzle. In general, perchlorate was detected in an average concentration of 0.5 µg/m<sup>2</sup> per motor. No comparison for day 1 between traps and plates can be established since the plates were not sampled. Results for day 2 were quite similar, with even less variation between samples. On the third and fourth days, we observed a few samples that present higher concentrations of perchlorate particle dispersed in the vicinity of the motor nozzle. On day 3 (Table 3), samples labelled W-17, 18, 19 and 20 present concentrations one or two orders of magnitude higher and are highlighted in red font in the table. It was decided to calculate the average concentration dispersed in two areas, the first one being nearest to the nozzle with a dimension of 20 m x 50 m (1000 m²), and the second one in the furthest 20 m x 150 m (3000 m²). On day 4, samples W-1 and W-20, located respectively at 12 and 62 m from the motor nozzle present higher concentrations of perchlorate, while other samples show lower levels than the first three days, with an average concentration of  $0.2~\mu g/m^2$  per motor.

Table 2: Water trap results, June 20<sup>th</sup> and 21<sup>st</sup>.

Water trap results							
					μg/m²		
	Perchlorate	Volume			per		
Sample	μg/L	(I)	μg/trap	μg/m <sup>2</sup>	motor		
Day 1, 2 motors, heavy rain							
W-1-AIM06-1	0.05	1.94	0.10	0.77	0.38		
W-2-AIM06-1	0.04	1.81	0.07	0.57	0.29		
W-3-AIM06-1	0.09	1.99	0.18	1.41	0.71		
W-4-AIM06-1	0.04	2.83	0.11	0.89	0.45		
W-5-AIM06-1	0.07	2.40	0.17	1.33	0.66		
W-6-AIM06-1	0.11	2.03	0.22	1.77	0.88		
W-7-AIM06-1	0.03	2.47	0.07	0.59	0.29		
W-8-AIM06-1	0.03	1.95	0.06	0.46	0.23		
W-9-AIM06-1	0.18	1.26	0.23	1.79	0.90		
W-10-AIM06-1	0.07	1.74	0.12	0.96	0.48		
W-11-AIM06-1	0.07	2.00	0.14	1.10	0.55		
W-12-AIM06-1	0.05	1.86	0.09	0.73	0.37		
W-13-AIM06-1	0.05	1.76	0.09	0.69	0.35		
W-14-AIM06-1	0.03	1.76	0.05	0.42	0.21		
W-15-AIM06-1	0.21	2.16	0.45	3.59	1.79		
W-16-AIM06-1	0.03	0.95	0.03	0.23	0.11		
Average					0.5		
St. Dev.					0.4		
	Day 2, 3 m						
W-1-AIM06-2	0.35	0.70	0.24	1.92	0.64		
W-2-AIM06-2	0.49	0.37	0.18	1.43	0.48		
W-3-AIM06-2	0.33	0.56	0.18	1.46	0.49		
W-4-AIM06-2	0.81	0.68	0.55	4.33	1.44		
W-5-AIM06-2	0.19	0.60	0.11	0.89	0.30		
W-6-AIM06-2	0.42	0.41	0.17	1.36	0.45		
W-7-AIM06-2	0.12	0.68	0.08	0.65	0.22		
W-8-AIM06-2	0.22	0.67	0.15	1.16	0.39		
W-9-AIM06-2	0.23	0.63	0.15	1.15	0.38		
W-10-AIM06-2	0.41	0.18	0.08	0.59	0.20		
W-11-AIM06-2	0.15	0.57	0.09	0.68	0.23		
W-12-AIM06-2	0.29	0.44	0.13	1.01	0.34		
W-13-AIM06-2	0.14	0.37	0.05	0.41	0.14		
W-14-AIM06-2	0.24	0.54	0.13	1.03	0.34		
W-15-AIM06-2	0.18	0.58	0.10	0.82	0.27		
W-16-AIM06-2	0.27	0.23	0.06	0.50	0.17		
W-17-AIM06-2	0.50	0.54	0.27	2.12	0.71		
W-18-AIM06-2	0.15	0.93	0.14	1.10	0.37		
W-19-AIM06-2	0.23	0.85	0.19	1.54	0.51		
Average					0.4		
St. Dev.					0.3		

Table 3: Water trap results, June 28th.

Water trap results								
Sample	Perchlorate μg/L	Volume (I)	μg/trap	μg/m²	μg/m² per motor	рН		
	Day 3, 4 motors, light rain							
W-1-AIM06-3	0.21	0.35	0.07	0.58	0.15	3		
W-2-AIM06-3	0.22	0.40	0.09	0.69	0.17	4		
W-3-AIM06-3	0.09	0.31	0.03	0.22	0.06	4		
W-4-AIM06-3	0.12	0.30	0.04	0.28	0.07	3		
W-5-AIM06-3	0.12	0.29	0.03	0.27	0.07	6		
W-6-AIM06-3	0.28	0.38	0.11	0.83	0.21	6		
W-7-AIM06-3	0.53	0.37	0.19	1.53	0.38	6		
W-8-AIM06-3	0.28	0.36	0.10	0.79	0.20	6		
W-9-AIM06-3	0.39	0.41	0.16	1.26	0.31	6		
W-10-AIM06-3	0.19	0.37	0.07	0.56	0.14	6		
W-11-AIM06-3	0.11	0.46	0.05	0.40	0.10	6		
W-12-AIM06-3	0.07	0.36	0.03	0.20	0.05	6		
W-13-AIM06-3	0.04	0.37	0.01	0.12	0.03	6		
W-14-AIM06-3	0.11	0.32	0.04	0.28	0.07	6		
W-15-AIM06-3	0.16	0.39	0.06	0.50	0.12	6		
W-16-AIM06-3	0.36	0.33	0.12	0.95	0.24	6		
Average (1 to 16)					0.20			
St. Dev. (1 to 16)					0.10			
W-17-AIM06-3	28.8	0.23	6.62	52.32	13.08	0		
W-18-AIM06-3	5.89	0.33	1.94	15.35	3.84	2		
W-19-AIM06-3	16.20	0.27	4.37	34.55	8.64	4		
W-20-AIM06-3	5.70	0.30	1.72	13.6	3.40	3		
Average (17 to 20)					7.24			
St. Dev. (17 to 20)					5.08			

*Table 4: Water trap results, June 29<sup>th</sup>.* 

Water trap results							
					μg/m²		
	Perchlorate	Volume			per		
Sample		(I)	ua/tran	ua/m²		<b>Д</b>	
Sample	μg/L	motors, no	μg/trap	μ	IIIOIOI	рп	
W-1-AIM06-4	4.51	0.60	2.68	21.20	3.53	3	
W-2-AIM06-4	0.10	0.87	0.09	0.68	0.11	4	
W-3-AIM06-4	0.08	0.68	0.05	0.43	0.07	5	
W-4-AIM06-4	0.00	0.56	0.03	0.43	0.07	5	
W-5-AIM06-4	0.20	0.51	0.11	0.80	0.13	6	
W-6-AIM06-4	0.20	0.43	0.15	1.15	0.19	6	
W-7-AIM06-4	0.29	0.40	0.13	0.91	0.15	6	
W-8-AIM06-4	0.12	0.50	0.06	0.47	0.13	6	
W-9-AIM06-4	0.12	0.35	0.10	0.77	0.13	6	
W-10-AIM06-4	0.09	0.55	0.05	0.39	0.06	6	
W-11-AIM06-4	0.13	0.60	0.08	0.62	0.10	6	
W-12-AIM06-4	0.05	0.49	0.02	0.19	0.03	6	
W-13-AIM06-4	0.02	0.81	0.02	0.13	0.02	6	
W-14-AIM06-4	0.14	0.61	0.09	0.67	0.11	6	
W-15-AIM06-4	0.23	0.74	0.17	1.35	0.22	6	
W-16-AIM06-4	0.09	0.67	0.06	0.48	0.08	6	
W-17-AIM06-4	0.42	0.23	0.10	0.76	0.13	6	
W-18-AIM06-4	0.74	0.82	0.61	4.81	0.80	4	
W-19-AIM06-4	0.30	0.54	0.16	1.27	0.21	3	
W-20-AIM06-4	2.09	0.61	1.27	10.04	1.67	3	
Average (2 to 19)					0.16		
St. Dev (2-19)					0.17		
W-21-AIM06-B	nd	field blank					
W-22-AIM06-B	nd	trip blank					

On the last two days of the trial, the pH of the water traps was recorded with pH paper to verify the influence of the hydrochloric acid plume. Lower pH values were measured in the area located between 12 and 62 m from the motor nozzles (Tables 3 and 4). The hydrochloric acid produced by the efficient combustion of perchlorate is preferentially deposited in this area. Solid particles will travel farther than gaseous emissions in soft wind conditions, and that is what we observed with detectable levels of perchlorate at 200 m from the motor nozzle. The dispersion area of solid perchlorate particles is wider than our sampling template since we do not reach the boundary zone, where no perchlorate was detected in our traps. This means that we underestimate the total amount of perchlorate dispersed. We observe a slight decrease of concentrations measured with distances, but the dispersion pattern is quite homogeneous with comparable levels at almost all sampling locations.

# 3.4 Witness plates results

Results for the second day of the trial on June 22<sup>nd</sup> are presented in Table 5, while the results for the third and fourth days of the trial are presented in Table 6.

*Table 5: Witness plates results for June* 22<sup>nd</sup>.

Day 2, 1 motor						
P0-ALL-AIM06-2	na	0.13				
P1-ALL-AIM06-2	2.66	0.20	0.52	0.13	0.13	
P2-ALL-AIM06-2	11.60	0.09	1.01	0.14	0.14	
P3-ALL-AIM06-2	4.21	0.26	1.08	0.12	0.12	
		Day 2, 1 mg	otor			
P0-ALL-AIM06-3	na	0.10				
P1-ALL-AIM06-3	na	0.05				
P2-ALL-AIM06-3	4.74	0.18	0.87	0.12	0.13	
P3-ALL-AIM06-3	3.92	0.19	0.76	0.08	0.08	
		Day 2, 1 mg	otor			
P0-ALL-AIM06-4	4.65	0.16	0.74	0.149	0.15	
P1-ALL-AIM06-4	2.55	0.16	0.41	0.103	0.10	
P2-ALL-AIM06-4	12.60	0.21	2.58	0.369	0.37	
P3-ALL-AIM06-4	5.15	0.19	0.99	0.110	0.11	
Day 2, average/per motor						
average					0.15	
standard deviation					0.09	
na = non available	na = non available					

In Table 5, results were not available for three samples, due to problems encountered with the analytical method at Environment Canada. The concentrations detected in water samples extracted from the wipes, as well as the volume of water used for the extraction, are reported. This allowed the calculation of the quantity of perchlorate in  $\mu g$  dispersed per row, and by dividing by the number of plates in each row (row 0=5 plates, row 1=4, row 2=7 and row 3=9), the quantity dispersed per square meter could be determined since each witness plate had a surface of  $1 \text{ m}^2$ . This allowed the calculation of the quantity dispersed per square meter per motor and, for the second day, it can be seen that most of the area sampled presented similar concentration ranges. A slight decrease in concentration with distance can be seen, with the exception of the row located at a distance of 100 m for the third motor, where the concentrations measured were twice the one in other rows. This might be explained by a motor that would have had a poorer combustion and spread more particles in the vicinity of the nozzle or else by the meteorological conditions. The high speed camera video allows the visual observation of particle emissions, especially in the sustain portion of the motor. The average concentration per square meter was calculated and this result presents an acceptable standard deviation. This suggests that

the static burning of three motors on day 2 generated the deposition of  $0.2 \,\mu\text{g/m}^2$  of perchlorate particles in a global area of  $4000 \,\text{m}^2$ .

*Table 6: Witness plate results for June 28<sup>th</sup> and 29<sup>th</sup>.* 

	Perchlorate	Volume			μg/m²	
Sample	μg/L	(1)	lug/row	ua/m²	per motor	
Sample μg/L (I) μg/row μg/m² per motor  Day 3, 1 motor						
P0-ALL-AIM06-5	1.11	0.26	0.29	0.06	0.06	
P1-ALL-AIM06-5	0.53	0.29	0.16	0.04	0.04	
P2-ALL-AIM06-5	1.53	0.20	0.30	0.04	0.04	
P3-ALL-AIM06-5	2.64	0.19	0.49	0.05	0.05	
		Day 3, 3 motor				
P0-ALL-AIM06-6	12.80	0.22	2.76	0.55	0.18	
P1-ALL-AIM06-6	39.00	0.17	6.59	1.65	0.55	
P2-ALL-AIM06-6	1.12	0.23	0.26	0.04	0.01	
P3-ALL-AIM06-6	0.88	0.16	0.14	0.02	0.01	
average					0.12	
standard deviation					0.18	
		Day 4, 1 motor				
P0-ALL-AIM06-7	1.09	0.22	0.24	0.05	0.05	
P1-ALL-AIM06-7	0.73	0.13	0.09	0.02	0.02	
P2-ALL-AIM06-7	1.03	0.13	0.14	0.02	0.02	
P3-ALL-AIM06-7	0.90	0.10	0.09	0.01	0.01	
		Day 4, 1 motor				
P0-ALL-AIM06-8	1.22	0.16	0.19	0.04	0.04	
P1-ALL-AIM06-8	0.39	0.18	0.07	0.02	0.02	
P2-ALL-AIM06-8	0.77	0.23	0.17	0.02	0.02	
P3-ALL-AIM06-8	0.60	0.14	0.08	0.01	0.01	
		Day 4, 2 motor				
P0-ALL-AIM06-9	matrix problems	0.18				
P1-ALL-AIM06-9	0.75	0.20	0.15	0.04	0.02	
P2-ALL-AIM06-9	2.1	0.28	0.59	0.08	0.04	
P3-ALL-AIM06-9	0.8	0.13	0.11	0.01	0.01	
average					0.02	
standard deviation					0.01	
Day 4, 2	motor: Results no	n available at the tin	ne of report	producti	on	
P-5-AIM06-B1	n.d.	laboratory blank				
P-5-AIM06-B2	n.d.	field blank				

Results for day 3 presented in Table 6 are fairly homogeneous and showed no trend with distance from the first motor nozzle. The second sampling after three motors showed higher concentrations for rows 0 and 1. On day 4, levels were relatively homogeneous and lower than for the other days, which could be attributed to the absence of light rain and the higher potential for particle loss. Results on day 3 are not normally distributed for the four set of samples with a standard deviation superior to the average concentration.

### 3.5 Surface soils results

Perchlorate was either not detected or detected at very low levels in all soil samples collected. Results indicated that soils could not be used effectively as a receptacle to monitor the deposition of perchlorate in such a study, levels deposited being far too small for effective detection.

### 3.6 Global results

The global results obtained from both media are presented in Table 7. In general, the quantities of perchlorate particles dispersed per square meter obtained with the use of witness plates are lower than those obtained with the use of water traps. This was expected, considering the higher risk of loosing particles on the metallic surface because of the wind. Further calculations were based on the water trap results. An average global quantity of perchlorate dispersed by motor was calculated using water trap results. If we exclude samples W-17 to W-20 for day three, we obtain an average dispersion of 1.3 mg per motor. If we include W17 to W20 results, we obtain an average dispersion of 2.4 mg per motor. Considering the high uncertainty attached to these values, we can assume that 2 mg of perchlorate particles are dispersed by each motor. MK-58 motors contain 47 kg of AP, this means that we have measured a dispersion of only 2 mg out of 47,000,000 mg, resulting in 4.25 x 10 <sup>-6</sup> % of perchlorate expelled per motor. This value is considered negligible especially during the actual flight of the missiles where 2 mg are dispersed over a distance of approximately 50 km.

Table 7: Global results.

Total quantity of AP dispersed							
Day	Water trap result average concentration μg/m²	Witness plate result average concentration μg/m²	Surface area impacted m <sup>2</sup>	Total AP dispersed (mg)			
1	0.5	nd	4000	2			
2	0.4	0.2	4000	1.6			
3	0.2	0.1	3000	0.6			
3	7	0.1	1000	7			
4	0.2	0.02	4000	0.8			

### 4 Conclusions

The present study is one of the first ever published on the assessment of the dispersion of perchlorate in the environment during normal burning conditions of tactical missile motors. Static firing was appropriate for this goal, since it keeps the residues in the smallest area possible. Even with static firing, we have seen that for medium range rockets such as the MK-58 motors, a sampling template of 4000 m² did not succeed in covering the overall dispersion of perchlorate. A larger sampling area that would have allowed the complete delineation of the AP particle plume would be needed to calculate a better estimate of AP dispersed. We underestimated the proportion of perchlorate emitted during the static firing of rockets motors. It would be interesting to repeat this experiment with a set-up that would allow reaching the boundary area impacted with perchlorate particles.

Another conclusion of this study is that the percentage of solid AP dispersed by the static firing of MK-58 motors is very small and quite homogeneous over our sampling template. The low dispersion of perchlorate particles was expected since perchlorate is a very good oxidizer, which leads to propellants that are well oxygen-balanced with high burn rate. The combustion process is, therefore, very efficient and almost all perchlorate is transformed into hydrochloric acid. If we assume that the perchlorate dispersed is underestimated by a factor of 10, we would still have a negligible weight to weight percentage of perchlorate dispersed of 4.25 x 10<sup>-5</sup>%. The solid propellant of MK-58 motors is representative of the various formulations used in modern APbased motors. Therefore, we may assume that the combustion efficiency of similar missiles should be very good, leading to a low dispersion of perchlorate particles. We have not included other contaminants of concern in the present study such as the anti-oxidant used in MK-58 propellant, phenyl-beta-naphtylamine. This latter compound is present at less than 1% by weight in the propellant but it might be considered as a health hazard since it has adverse toxicological impacts. Considering the low levels of perchlorate dispersed, we do not foresee that measurable quantities of this analyte could have been detected in any sample. However, it may burn less efficiently than the oxidizer and future trials might include it as an analyte.

The range of the MK-58 motor or the AIM-7 rocket is approximately 50 km. So, at the most, if we include a factor of 10 times over what we found, 20 mg of perchlorate would be dispersed in the environment over a 50 km wide area at a high altitude. This means that the use of AIM-7 in live-fire training does not contribute to any measurable impact in terms of perchlorate deposition/accumulation. This is also probably true for other AP-based rockets. This is not true for air-to-ground launches, when the rocket may reach the target before completely burning its propellant. In these circumstances, the remaining propellant might be either consumed in the detonation fireball upon impact, or else be dispersed in the environment if the detonation process is incomplete or inefficient.

Accumulation of perchlorate on test sites where static burning of AP-based rockets are conducted might happen, but considering the small impact per motor, the frequency of static firings would have to be quite high to reach levels of concern. Caution should be maintained for such sites, especially if frequent static firings of AP-based motors are conducted on a specific test site. An approximation of the concentrations of perchlorate could be estimated using this study but sampling of the surrounding soils and groundwater should also be conducted.

Another potential measurable source of perchlorate in the environment could be very large solid rocket motors, such as the booster used for the Space Shuttle. For example, approximately 70 percent of the weight of the solid propellant in the Space Shuttle boosters consists of ammonium perchlorate (Ref. 23). Each pair of Shuttle motors uses about 1.7 million pounds of ammonium perchlorate; thus, the Space Shuttle is the largest user of ammonium perchlorate and repeated use of the same site for vertical launches might present a risk for perchlorate accumulation.

The main conclusion of the present study is that the use of AP-based rocket motors in live-fire training by our troops does not represent a high potential source for perchlorate in the environment. Most of the propellant is consumed during the burning and the remaining small unburned portion is dispersed in the wide trajectory of the rocket.

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# List of symbols/abbreviations/acronyms/initialisms

ADATS Air Defense Anti-Tank System

AP Ammonium Perchlorate

CTPB Carboxy Terminated PolyButadiene

DGAEPM Director General of Aerospace and Engineering Program Management

DND Department of National Defence

DRDC Defence Research & Development Canada

DRDKIM Director Research and Development Knowledge and Information

Management

EPA Environmental Protection Agency

ER Environmental Restoration

FPS Frame Per Second

HTPB Hydroxy Terminated Polybutadiene

kN Kilo Newton

METC Munitions Experimental Test Center

PBNA Phenyl-beta-naphtylamine

RDX Research Development Explosive

R&D Research & Development

SERDP Strategic Environmental R&D Program

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Static firings of AIM-7 rocket motors were conducted in June 2006 at the DRDC Valcartier test site to measure the environmental dispersion of ammonium perchlorate (AP) during normal burning conditions. The recent stringent threshold criterion for perchlorate in drinking water dictates that the sources of this contaminant be better defined. The AIM-7 Sparrow missile, which is propelled by an MK-58 AP-based motor, has been the main medium range air-to-air missile used by both the US and Canadian fighter jets for many years. Fifteen MK-58 motors were obtained for this study from the Canadian inventory and statically fired on a test bench to measure the residues that were expelled during combustion. The field set-up to catch perchlorate particles was based on a combination of aluminium witness plates and aqueous traps located in the exhaust plume area. The tests were conducted over four days and sampling was achieved after the burning of two, three, four and six motors. The set-up allowed the collection of a large portion of the exhaust plume, and perchlorate was detected in most of the samples collected, due to the use of a very sensitive analytical method. The results obtained were consistent and reproducible, and it was estimated that only 2 mg of perchlorate was deposited per MK-58 motor. Considering the motors flight range, it is concluded that their use in live fire training does not contribute to the accumulation of perchlorate in the environment.

Des mises à feu statiques de moteurs-fusées AIM-7 ont été réalisées en juin 2006 sur le site d'essais de RDDC Valcartier, pour mesurer la dispersion environnementale du perchlorate d'ammonium (PA) dans des conditions normales de brûlage. Le critère de seuil rigoureux émis récemment concernant le perchlorate dans l'eau potable implique de déterminer les sources potentielles de ce contaminant. Le missile Sparrow AIM-7, propulsé par un moteur MK-58 à base de PA, est le principal missile air-air de moyenne portée utilisé sur les avions de chasse américains et canadiens depuis de nombreuses années. Quinze moteurs MK-58 provenant de l'inventaire Canadien ont été lancés de façon statique avec un banc d'essai fixe pour mesurer les résidus déposés durant la combustion. Les essais ont été réalisés sur une durée de quatre jours et l'échantillonnage a été effectué après la mise à feu statique de deux, trois, quatre et six moteurs MK-58. La stratégie de collection des particules a été élaborée en combinant des plaques témoins en aluminium et des trappes d'eau dispersées dans la zone du panache de fumée. Cela a permis la récolte d'une large portion du panache de dépôt et le perchlorate a été détecté dans la plupart des échantillons compte tenu d'une méthode d'analyse très sensible. Les résultats obtenus sont cohérents et reproductibles et il a été estimé que chaque moteur MK-58 dépose seulement 2 mg de perchlorate. Considérant la distance de vol de ces moteurs, on peut conclure que l'utilisation de ceux-ci dans l'entraînement en tir réel ne contribue pas de façon significative à l'accumulation de perchlorate dans l'environnement.

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