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THE EVAPORATION AND GASEOUS DISPERSION OF LARGE-SCALE RELEASES OF NITROGEN TETROXIDE*

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ABSTRACT

(3-5 cubic m)

A series of large-scale spills (3-5 m^3) of nitrogen tetroxide (N_2O_4) was performed at the Nevada Test Site (NTS) for the Air Force Engineering and Services Laboratory, Environics Division. The purpose of these experiments was to study the evaporation rates and heavy gas dispersion aspects of realistic size releases of N_2O_4, a rocket fuel oxidizer for the Titan II missile system. The Titan II complexes are currently being decommissioned, resulting in an increase in the overland transportation of this toxic material. The normal boiling point for the liquid N_2O_4 is ~~21°C~~ 21 deg C, hence it evaporates rapidly when spilled onto warm soil and quickly dissociates into nitrogen dioxide (NO_2) as it mixes with air. The NO_2 is highly toxic with an ACGIH threshold limit value 3 parts per million (ppm).

The design of the spill facility and the diagnostics are described. Some typical results for one of the spills are presented. The data include the atmospheric boundary layer conditions in effect during the spill, the spill area heat flux and vapor temperature, the vapor flux measured at 25 meters (m) downwind, and the NO_2 gas concentration contours at 785 m.

A comparison of the measured NO_2 concentration and that calculated with the Air Force Ocean Breeze/Dry Gulch (OB/DG) model(2) is discussed.

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INTRODUCTION

The Lawrence Livermore National Laboratory (LLNL) conducted a series of large-scale (3-5 m^3) N_2O_4 spill tests for the U.S. Air Force during the fall of 1983. The purpose of the test series was to determine the heavy gas dispersion aspects and source strength characteristics of large N_2O_4 spills, and to provide N_2O_4 spills for the evaluation of a Portable Foam Vapor Suppression System (PFVSS). The dispersion and source strength tests were under the sponsorship and direction of the Engineering and Services Center, Tyndall AFB. The PFVSS effort was under the direction of the Ogden Air Logistics Center, Hill AFB, with support from USAF Space Division and the Strategic Air Command. The tests were performed at the U.S. Department of Energy (DOE) Nevada Test Site (NTS) under the jurisdiction of the DOE Nevada Operations Office (NVO).

The N_2O_4 tests were the fifth in a continuing program of hazardous materials spill tests conducted by LLNL and were code-named the Eagle series. There were a total of six N_2O_4 spill tests. Four tests were for the purpose of dispersion and source strength studies (Eagles 1, 2, 3, and 6) and two tests for evaluation of the PFVSS (Eagles 4 and 5). The Eagle series was conducted in conjunction with a series of four ammonia (NH_3) spill tests (Desert Tortoise series) at considerable savings to both sponsors. These back-to-back series began with NH_3 on August 12, 1983, followed by the change-over to N_2O_4 in mid-September. The Eagle series tests were performed between September 17 and November 30.

The primary purpose of this paper is to present a representative sample of the results that are available in the Eagle Series Data Report.[1] A brief description of the experiment and diagnostics is included. Some typical results of the Eagle 3 spill are presented. In addition, a comparison of the measured NO_2 concentrations at 785 and 2800 m with the predictions of the Air Force OB/DG model is also presented.

EXPERIMENT DESCRIPTION

The two primary components of the temporary N_2O_4 spill facility were an Air Force-supplied R-16 tanker and a LLNL N_2 gas tube trailer. A layout of the spill facility is shown in Fig. 1. The N_2 tube trailer was used to pressurize the R-16 tanker and force the N_2O_4 through the spill pipe to the spill point, to provide purge gas for cleansing the piping system after each spill, and to provide gas pressure for the pneumatically operated valves.

A typical spill test sequence would proceed as follows. On days with favorable weather forecasts, the diagnostic system would be checked for satisfactory operation, and the spill area would be cleared of all personnel except for the arming team. Members of the arming team would open the manual valve on the N_2 tube trailer, set the pressure control valve to the desired drive gas pressure and open the manual valves on the R-16 tanker. The arming team would then leave the area. All further spill operations were conducted remotely.

When the wind speed and direction were within the designated acceptable spill conditions, the R-16 tanker was pressurized and the spill was initiated. A real-time display of the volume of N_2O_4 spilled as a function of time was provided by the command and control data recording system (CCDRS) located about 1 km upwind. When the desired amount of N_2O_4 had been spilled, the spill was terminated. After the vapor cloud had cleared the downwind array, the pressure in the R-16 would be relieved and the disarming team would then enter the area and close the manual valves on the R-16 tanker to secure the facility.

The N_2O_4 was delivered to the spill area by a 30-m long, 7.62-cm diameter (3-in) PVC pipe where it was distributed in two different ways. The single-exit, confined spill configuration (Fig. 2a) was for the purpose of studying evaporation rates as a function of liquid pool depth and wind speed. The multi-exit, unconfined spill configuration (Fig. 2b) was designed to distribute the N_2O_4 over a large area in order that it evaporate as quickly as it was spilled. This would produce a large source of N_2O_4 vapor for the dispersion studies.

Numerous measurements were made in the area of the spill. The temperature of the N_2O_4 just prior to its exit from the spill pipe was recorded. Three heat-flux sensors were placed just below the surface of the soil at different locations. A thermocouple rake assembly was also installed in the spill area for the purpose of determining the temperature gradient within the liquid for the confined spills, and within the initial vapor layer of the unconfined spills. One thermocouple was at ground level, and the second and third at heights of 2 and 4 cm, respectively. Provision was also made for measuring the depth (pressure head) of the liquid N_2O_4 during the confined spills.

For tests Eagle 1 through 3 and Eagle 6, the N_2O_4 was spilled directly onto the ground, whereas for Eagles 4 and 5 a PVC plastic liner was used to help contain the liquid. Although the lakebed playa surface was clay-like and known for its impermeability to water, the N_2O_4 soaked into it quite readily. In many cases it actually caused the surface to heave up several inches. There was considerable outgassing from the surface for several hours after the spill was terminated. After each of spills Eagles 1 thru 3 and Eagle 6, safety considerations required spraying 100-1000 gallons of water to dilute the N_2O_4 absorbed by the ground to the point that its vapors were reduced to acceptable levels.

In addition to the spill area measurements, atmospheric boundary layer, wind field, vapor cloud temperature and concentration, and surface heat flux measurements were also made using an extensive diagnostic system developed by LLNL. There are three main array systems: the meteorological array, the mass flux array, and the dispersion array. The locations of these various arrays, along with the positions of the camera stations, are shown in Fig. 3.

The meteorological array consisted of nine two-axis, cup-and-vane anemometers (all at a height of 2 m), plus a 20-m tall tower located directly upwind of the spill area. The locations of the nine wind field stations are shown in Fig. 3. Wind speed and direction at each station were averaged for 10 sec, and the results, plus the standard deviation of direction for the same period, were transmitted back to the CCDRS trailer. The wind field data were displayed in real time and were the primary information used to determine the optimum time for the spill.

The meteorological boundary layer data were obtained from measurements mounted on a 20-m tower located 50 m directly upwind of the spill point (Fig. 3). This tower was outfitted with four temperature gauges and three Gill bivane anemometers. This station also measured the ground heat flux. Humidity data and local barometric pressure were obtained from the NTS Weather Support Group.

A mass flux array was employed to determine the evaporation rate, or source strength, of the N_2O_4. This was to be accomplished by measuring the N_2O_4 concentration, vapor cloud temperature and velocity as it passed through the array. The N_2O_4/NO_2 ratio would be determined using a well-documented equilibrium reaction rate constant.[3,4] The cloud temperature data is required for calculations of the rate constant and the conversion from concentration to mass density. The product of the mass density and velocity integrated over the vapor cloud cross-section yields the total mass flux passing through the array at any instant. If the entire cloud is "captured" by the array, this mass flux should be equivalent to the vapor source strength of the spilled N_2O_4.

The mass flux array was located 25 m downwind of the spill area and consisted of seven gas stations and two wind-speed stations. The centerline station was a 10 m tall tower outfitted with three bivane anemometers, three LLNL infrared (IR) gas sensors, and three thermocouples. The three anemometers were located at heights of 1.3, 3, and 6 m for the entire Eagle series. The IR gas sensors and thermocouples were located at different heights for each test. These stations had 6 m tall masts and each was outfitted with three LLNL IR gas sensors and three thermocouples. There were six additional stations located at 5 m intervals to either side of the centerline station (three to each side).

A detailed description of the LLNL IR gas sensor is given in Ref. 5. The sensor produces a signal proportional to the molecular absorption of IR radiation by the N_2O_4 vapors as they pass through the 15 cm sample region. The sensor was calibrated by using known concentrations of N_2O_4. The sensor was originally designed for the detection of liquefied natural gas (LNG) vapors and was not optimized for the Eagle series experiments to separately detect

N_2O_4 and NO_2. The species of most interest here was NO_2, however when it was discovered that the IR sensor was able to effectively detect N_2O_4 vapors without any modifications, the cost savings dictated that this approach be taken. Unfortunately, this required placing the sensors very close to the source, which resulted in severe acid damage to several of them.

Two wind speed stations were located in the mass flux array at ± 7.5 m to either side of the array centerline. These were similar to the wind field stations described earlier, except that these anemometers were placed at a height of 1 m above the ground. The purpose of these measurements was to determine if there were spatial wind speed variations as the vapor cloud passed through the array.

The dispersion array consisted of five 10 m towers located 785 m downwind of the spill area (see Fig. 3). The purpose of this array of sensors was to record the extent of the vertical cross-section of the NO_2 vapors during each spill. All the towers had three NO_2 gas sensors and three thermocouples located at heights of 1, 3.5, and 8.5 m above the ground. The towers were separated by a distance of 100 m. The NO_2 gas sensors were furnished by the Shuttle Activation Task Force, Vandenburg AFB for use during the Eagle series. All instruments were capable of full-scale measurements of 0-5, 0-50, and 0-500 ppm concentrations.

In addition to the gas sensors mounted on the towers of the 25 and 785 m arrays, two portable NO_2 gas sensors were also used during the tests. These sensors used an electro-chemical transducer, and drew the gas sample through a short (approx. 0.5 m) tube located about 15 cm above the ground. Both sensors had a maximum concentration range of 10 ppm NO_2, and were refurbished and recalibrated by the manufacturer just prior to the Eagle test series. These sensor were used to obtain data at 2800 m during the spill tests, and to monitor the CCDRS trailer park and the Frenchman Flat access roads overnight after the spills.

Photographic and video coverage of Eagles 1-3 were provided by LLNL. Photographic coverage of the PFVSS tests were the responsibility of Hill AFB. There was only video coverage of the Eagle 6 spill. The LLNL cameras were located as shown in Fig. 3. There were a total of five cameras: two motion picture cameras and three programmable framing cameras. All cameras were remotely controlled and began operating at the time the spill valve was opened. The programmable cameras provided coverage for up to 30 min at differing framing rates. The motion picture coverage was for a duration of about 20 min. Black and white video coverage was provided by a TV camera located 20 m directly upwind of the spill area. This camera was equipped with a remote zoom, pan and tilt capability, and was also used to monitor the facility arming and disarming procedure.

The control of the spills and the data acquisition and storage was all performed in the CCDRS trailer located at about 1 km from the spill point (see Fig. 3). This system utilizes UHF radio telemetry for command and data transmission and is designed to acquire data from sensors distributed over an area with a diameter of up to 10 miles[6]. All of the remote data acquisition stations and sensors are battery-powered, portable, gas-tight, and ruggedized. Batteries are recharged by solar cells. This network of 24 stations acquired data from up to 270 channels at a rate of one sample per second for the gas and control stations and one sample per 10 sec for the wind-field stations.

After each test, raw data are converted to fully calibrated data sets. These reduced data are written to an ASCII magnetic tape and transferred to the LLNL Computation Center for archival preservation. The data base tables are stored on an off-line mass storage system and are readily available for analysis.

EAGLE 3 PRELIMINARY RESULTS

As a typical example of the data contained in Ref. 1, some of the results of the Eagle 3 spill will be presented and discussed. A summary of the Eagle 3 spill parameters is given in Table I. This was the largest spill volume of all the Eagle series tests and was conducted under nearly ideal atmospheric conditions. The vapor cloud traveled directly down the array centerline producing NO_2 concentrations in excess of 500 ppm at 785 m. One of the portable NO_2 sensors located on the array centerline at 2800 m recorded a peak concentration of 9 ppm.

TABLE I.

EAGLE 3 EXPERIMENT SUMMARY:

Date:	7 October 1983
Time:	4:48 PDT
Test Objective:	Dispersion data
Spill Configuration:	Multi-exit, unconfined
Spill Volume:	4.2 m^3 (6090 kg)
Average Spill Rate:	1.4 m^3/min (2030 kg/min)
Spill Duration:	188 sec
N_2O_4 Spill Temperature:	19°C
Prespill Ground Temperature:	27°C
Average Wind Speed:	3.14 m/sec
Average Wind Variability:	$\sigma_\theta = 13.2°$

As the liquid N_2O_4 spilled, it was observed to evaporatively cool to its freezing point. This is believed to be the result of the poor thermal conductivity properties of the lakebed soil. The vapor temperature at a height of 2 cm and located approximately 1 m from one of the spill points is shown in Fig. 4. The normal freezing point for N_2O_4 is -12.2°C. One would expect this behavior of the N_2O_4 if it were allowed to pool. However, examination of the video tapes showed that very little of the N_2O_4 actually formed liquid pools. It either evaporated or was absorbed into the ground.

Estimates of the source strength can be made several ways. If we assume that all of the N_2O_4 evaporatively cools from 19°C to -12°C as soon as it is spilled, this would produce an evaporation rate of 240 kg/min. This calculation neglects all other sources of heat such as from the ground, air, or sun. This result (240 kg/min) is an upper bound for this particular contribution to the total evaporation process since it is unlikely that the total spill volume was cooled to this degree for the entire duration of the spill. The total evaporation process (source strength) depends on three main heat sources; the liquid internal energy (evaporative cooling), the ground heat flux, and the heat flux from above (air and sun).

A second estimate involves the ground heat flux as measured directly below the soil surface (Fig. 5). The sign convention for the ground heat flux is such that a positive value represents heat flowing <u>into</u> the ground. The drop in heat flux during the spill was much less than expected. Assuming that the multi-exit spill system distributed the N_2O_4 uniformly over a 20 m diameter area, and that it evaporated as fast as it was spilled, would require a total heat flux of about 50 kWatt/m^2. We see from Fig. 5 that the peak measured ground heat flux is about 100 times less than this amount. Clearly the N_2O_4 did not evaporate as quickly as it was spilled. If one assumes a uniform heat flux of 0.50 kWatt/m^2 over the 20 m diameter area, the resulting source strength is calculated to be 23 kg/min. This value (23 kg/min) is considered to be a minimum estimate of the source strength since it does not include the other sources of heat. The actual source strength would consist of the sum of the ground heat flux component, the sun and air heat addition component, and internal energy component mentioned earlier.

As previously discussed, the primary purpose of the mass flux array was to determine the source strength of each spill. This was to be accomplished by measuring the vapor density and velocity as it passed through the 21 gas sensors located in the vertical plane 15 m downwind of the spill area. The mass flux ($\dot{m}$) at any instant in time is calculated by integrating the product of the density and velocity over the entire cross-section of the vapor cloud, i.e.,

$$\dot{m} = {}_A\int \rho u \, dA \tag{1}$$

where ρ is the density, u is the vapor velocity normal to the array plane, and A is the cloud cross-sectional area. The summation of the instantaneous mass flux ($\dot{m}$) over the entire vaporization period should equal the amount spilled. This total mass evaporation calculation assumes no N_2O_4 is lost due to permeation into the ground.

It became immediately obvious upon examination of the Eagle 1 spill results that something other than N_2O_4 and/or NO_2 vapors were present in the vapor cloud. The LLNL IR sensor detects molecular absorption in four different spectral regions. For mixtures of N_2O_4 and NO_2 vapors, two the mist formation reaction will tend to increase the N_2O_4 dissociation and reduce the N_2O_4 concentrations at the 25 m array. Furthermore, since the IR gas sensors were not calibrated for HNO_3 mists, they can only produce estimates of the N_2O_4 vapor content of the cloud. That portion of the total mass flux due to the HNO_3 mist can only be inferred by subtracting the total vapor flux, summed over the entire spill, from the total mass spilled. While this is felt to be a worthwhile calculation, it does not give us the magnitude of the source strength.

The effect of the HNO_3 and NO products on the downwind dispersion is not known at this time. Nitric oxide (Eq. (2)) combines with molecular oxygen (O_2) to form NO_2 at a rate which is dependent on the O_2 concentration and the square of the NO concentration. At very low NO concentrations the oxidation rate is slow (at 10 ppm NO, seven hours are required for 50% oxidation). However, at a concentration of 1% NO, 50% oxidation is achieved in about 24 sec. The eventual downwind products of the HNO_3 mist are not known due to the highly reactive nature of this substance.

As a result of the HNO_3 mist, the N_2O_4 concentrations presented here and in the Eagle Series Data Report[1] pertain only to the vapor portion of the cloud. The IR gas sensor data was processed assuming the mist attenuation to be equal in both the signal and reference channels.

Nitrogen tetroxide concentration contours in the vertical plane of the 25 m array are shown in Fig. 6. These data represent the N_2O_4 concentration distribution across the 25 m array 90 sec after the beginning of the spill. The contours are as they would appear to an observer at the spill area looking in the downwind direction. The time and concentration levels (% vol) are indicated on each plot.

The contour routine assumes a linear interpolation between the concentration values recorded at each sensor location. In order that the contours not close below the lower level of sensors, extrapolations of the data were required. Two techniques were used to extrapolate the vertical concentration data to the ground level. If the middle height concentration at a station was less than the lower height value, the ground level concentration was determined

(10 And 12 missing)

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COMPARISON WITH THE OB/DG MODEL

The primary purpose of the Eagle test series was to demonstrate the heavy gas dispersion aspects of N_2O_4 vapors. The downwind reduction in concentration for the dispersion of a heavy gas is not as extensive as for a trace or neutral gas. For heavy gas dispersion, the size of the hazardous corridor is correspondingly much greater. In late 1960, the Air Force conducted a series of dispersion tests[2] at Cape Kennedy, Florida (Ocean Breeze) and at Vandenburg AFB, California (Dry Gulch). These tests involved the release and detection of a zinc sulfide tracer. There were a total of 185 tests performed under a wide range of atmospheric conditions. All of the data of the OB/DG tests were normalized and correlated to a simple diffusion prediction equation, the OB/DG model. This simple model predicted 75% of the cases to within a factor of two of the measured values. The model expresses the peak inhalation (1.5 m high) NO_2 concentration C_p (ppm) as a function of source strength Q (kg/min), the downwind distance X (m), the standard deviation in the horizontal wind direction σ_θ (degrees), and the temperature difference ΔT (°C) of the atmospheric boundary layer between the heights of 16.5 m (54 ft) and 1.83 m (6 ft), i.e.,

$$C_p = 3.535\, Q\, X^{-1.96} (1.8\, \Delta T + 10)^{4.33}\, \sigma_\theta^{-0.506} . \qquad (2)$$

We will now compare the results of the Eagle 3 test with the OB/DG prediction. Our first real problem is in defining the Eagle 3 source strength, for at this time, we can only place limits on the source strength. We know from the heat flux data and the observed source strength from the vapor flux calculations that the evaporation rate must certainly be greater than 23 kg/min. We also know that the maximum possible source strength must be less than the spill rate of 2030 kg/min. The σ_θ for Eagle 3 (13.2°) was averaged over 10 sec intervals, whereas those used in formulating the OB/DG model were averaged over 15 sec intervals. We will use the ΔT difference between the heights of 16.19 and 2.46 m (0.5°C). The results of the OB/DG concentration predictions at 785 and 2800 m for the minimum (23 kg/min) and maximum (2030 kg/min) possible Eagle 3 source strengths are given in Table II. Also shown in Table II are the peak measured concentrations at these locations. The OB/DG model results are very sensitive to the choice of ΔT and σ_θ. Future data analysis must resolve the effect of the different heights on ΔT and the different averaging period on σ_θ. However, it is felt that the calculated results of Table II are accurate to within ± 30%. It appears that the OB/DG model underestimates the downwind concentration distribution of the Eagle 3 spill, though the degree of the error at 2800 m is not clear due to the small number of measurements.

TABLE II.

COMPARISON OF EAGLE 3 NO_2 DATA AND OB/DG PREDICTIONS.

Downwind distance (m)	OB/DG Predictions (ppm)		Eagle 3 concentration measurements (ppm)
	Q = 23 kg/min	Q = 2030 kg/min	
785	1.5	130	> 500
2800	0.12	10.6	≥ 9*

* may not have been on cloud centerline

SUMMARY

A series of six N_2O_4 spills (Eagle series) was conducted at the Frenchman Flat area of the DOE Nevada Test Site in 1983. Four of these tests (Eagles 1-3, Eagle 6) were for the purpose of studying the source strength characteristics and heavy gas dispersion aspects of N_2O_4 spills on the ground. Two of these tests (Eagles 4 and 5) were for the purpose of evaluation of a portable foam vapor suppression system. The tests were performed between September 17, 1983 and October 30, 1983 and involved a total of 16 m^3 (4200 gal) of N_2O_4.

This paper has briefly described the experiments and shown some of the preliminary results of one of the tests. Future efforts will include a more thorough analysis of the spill area and mass flux data in order to better quantify the magnitude of the source strength. This work will also include a study of the HNO_3 mist formation process and the possible impact it may have on the measured results.

Once the source strength is better understood, a rigorous comparison of all of the 785 and 2800 m NO_2 data with the OB/DG predictions will be performed. Comparison with other more sophisticated models will also be conducted if time and resources allow.

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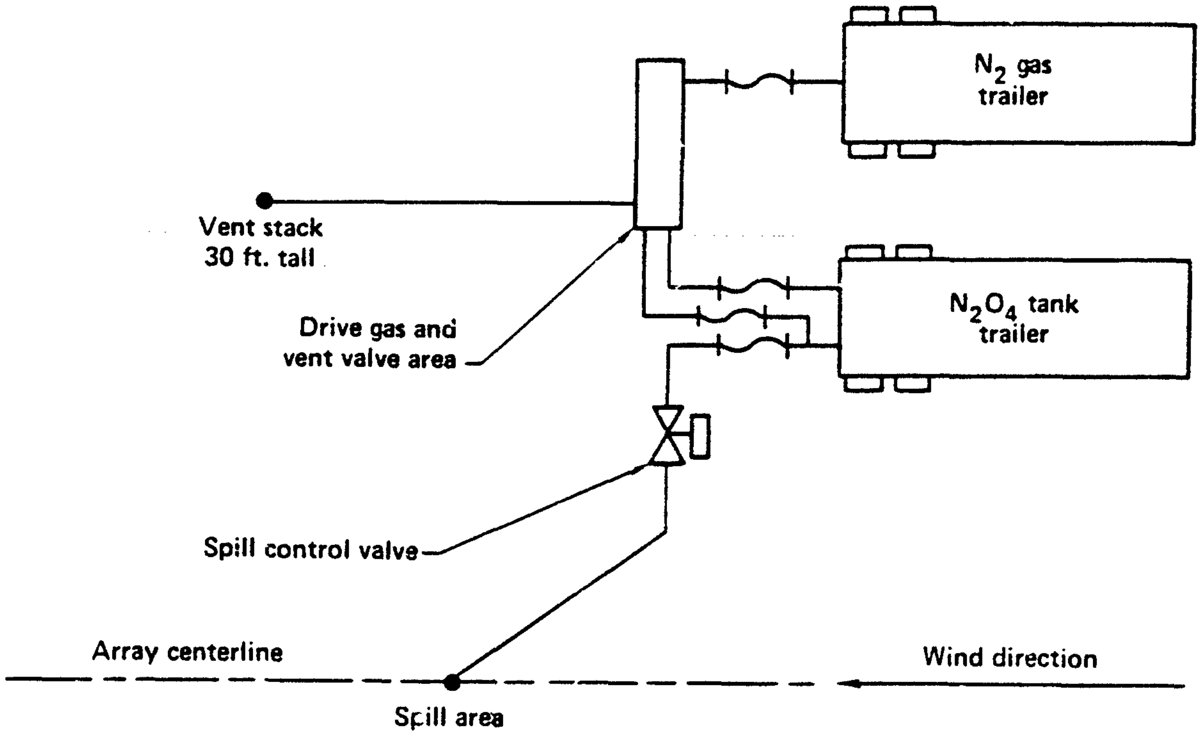
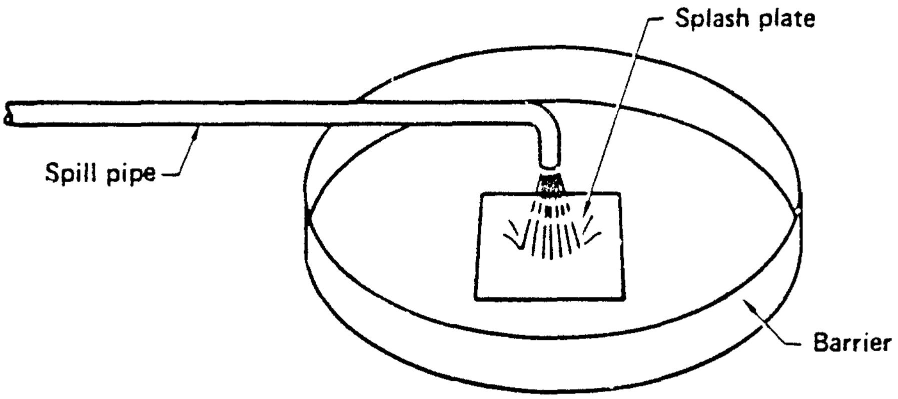
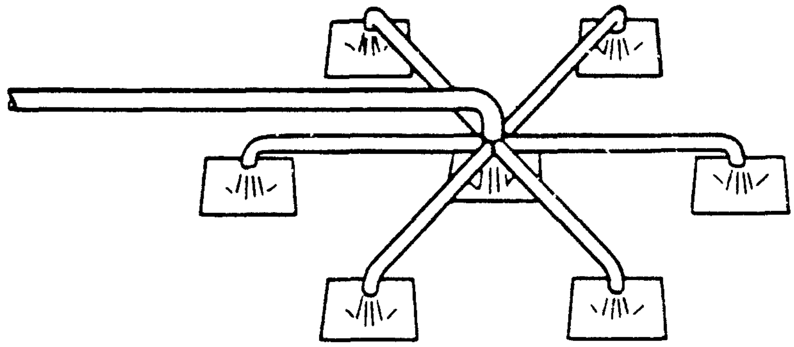


Fig. 1. Spill Facility Site Layout.



(a) Single-exit confined



(b) Multi-exit unconfined

Fig. 2. Eagle Series Spill Configurations.

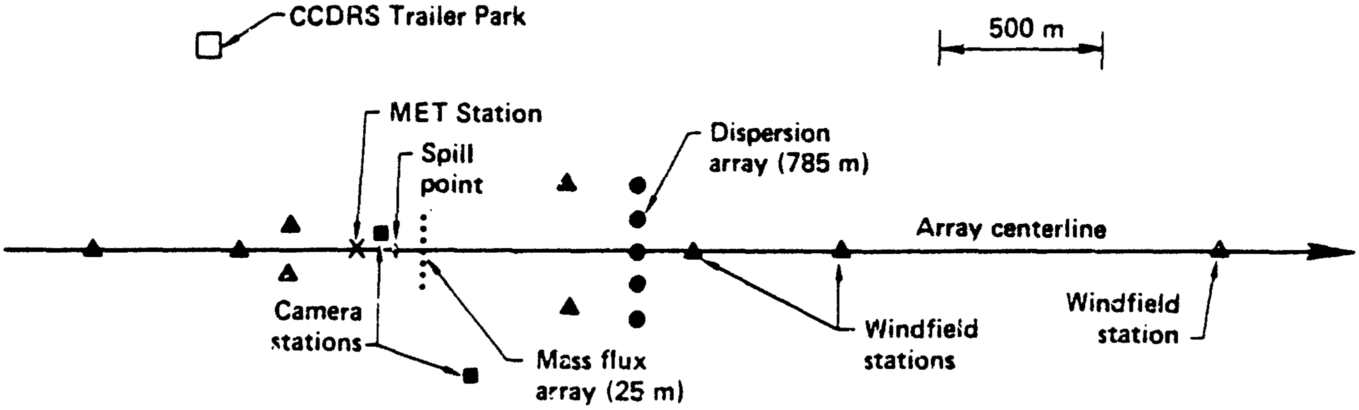


Fig. 3. The Eagle Series Diagnostic Array.

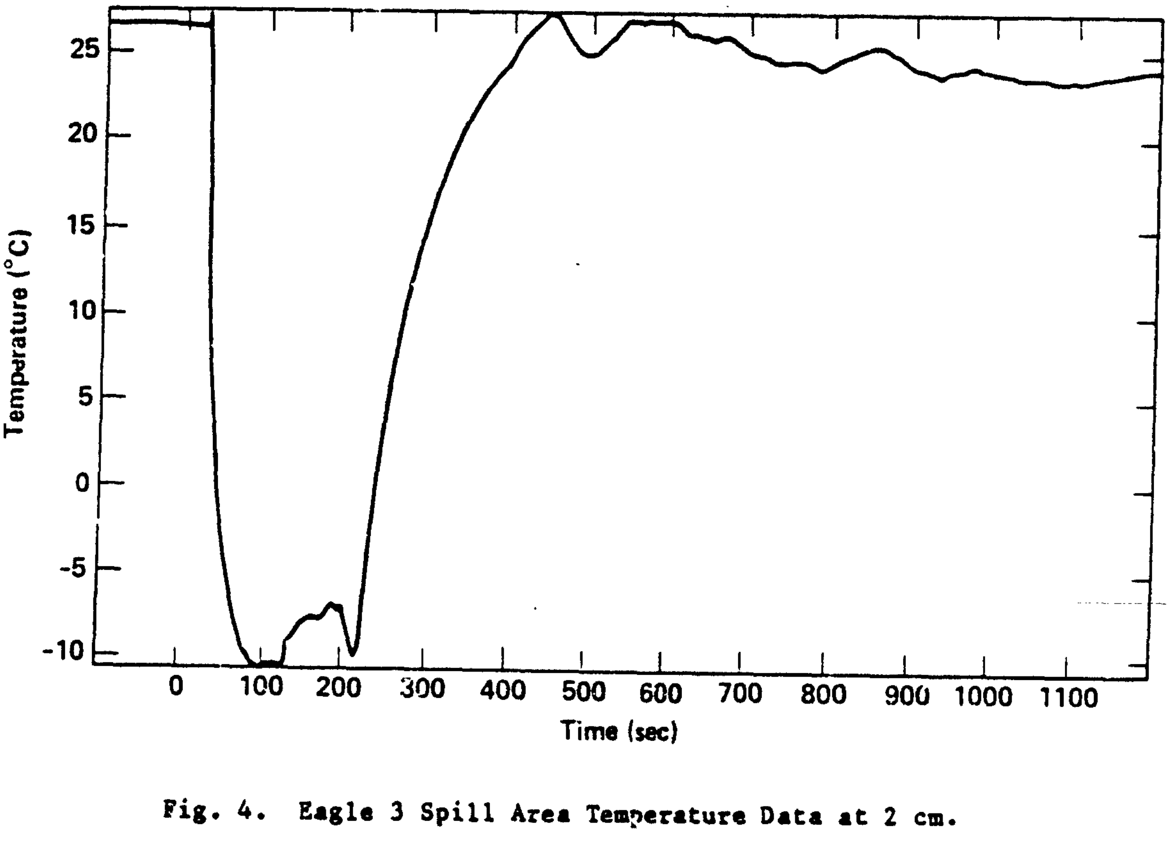


Fig. 4. Eagle 3 Spill Area Temperature Data at 2 cm.

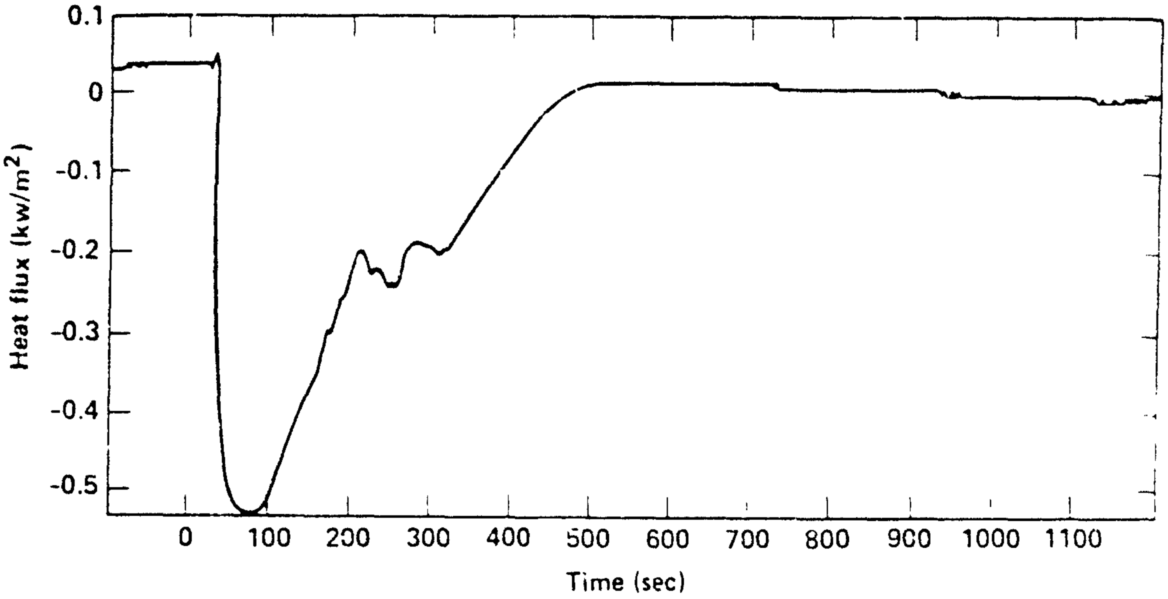


Fig. 5. Eagle 3 Spill Area Heat Flux Data.

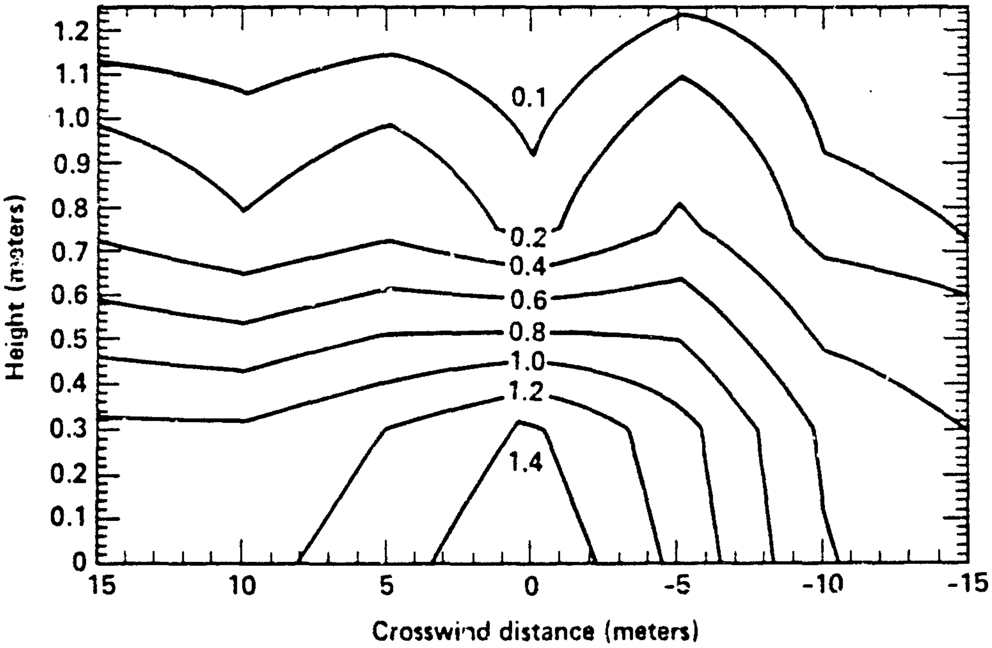


Fig. 6. Eagle 3 Crosswind Concentration Contours at 25 m.

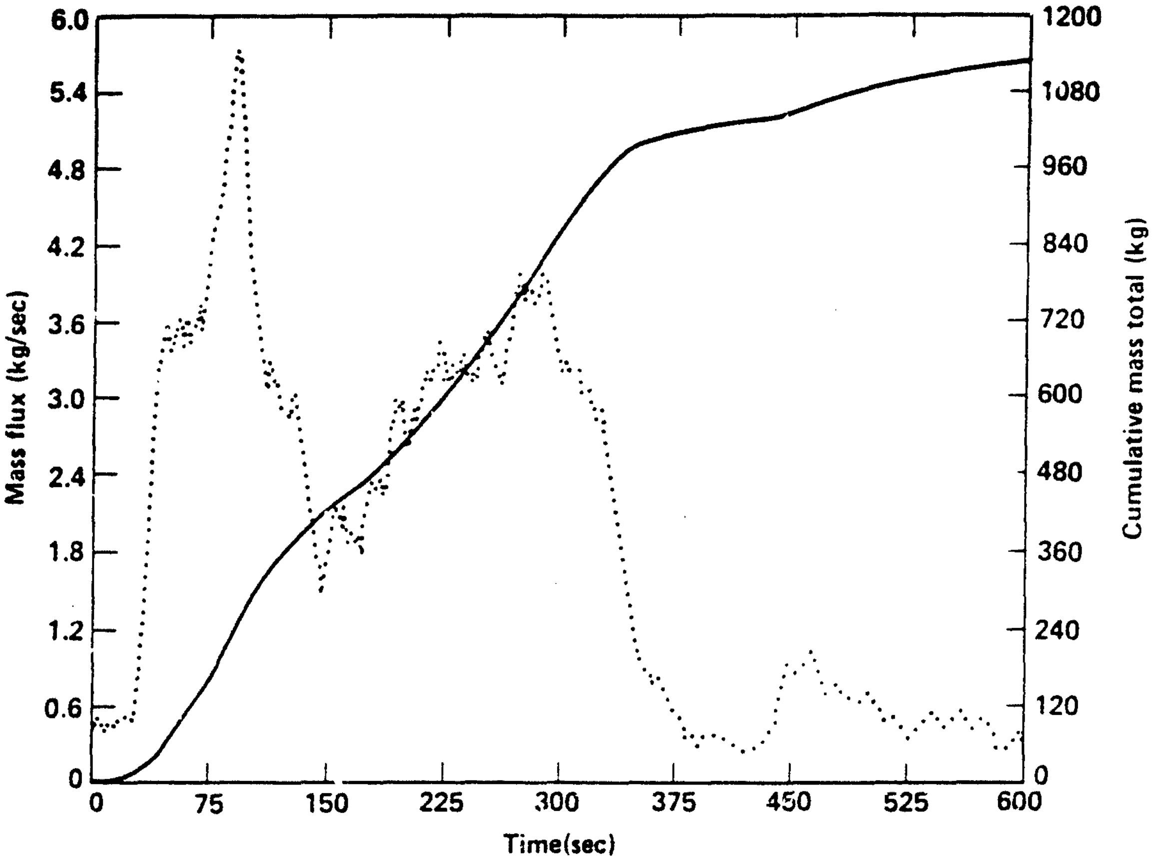


Fig. 7. Eagle 3 Vapor Flux Calculation Results.

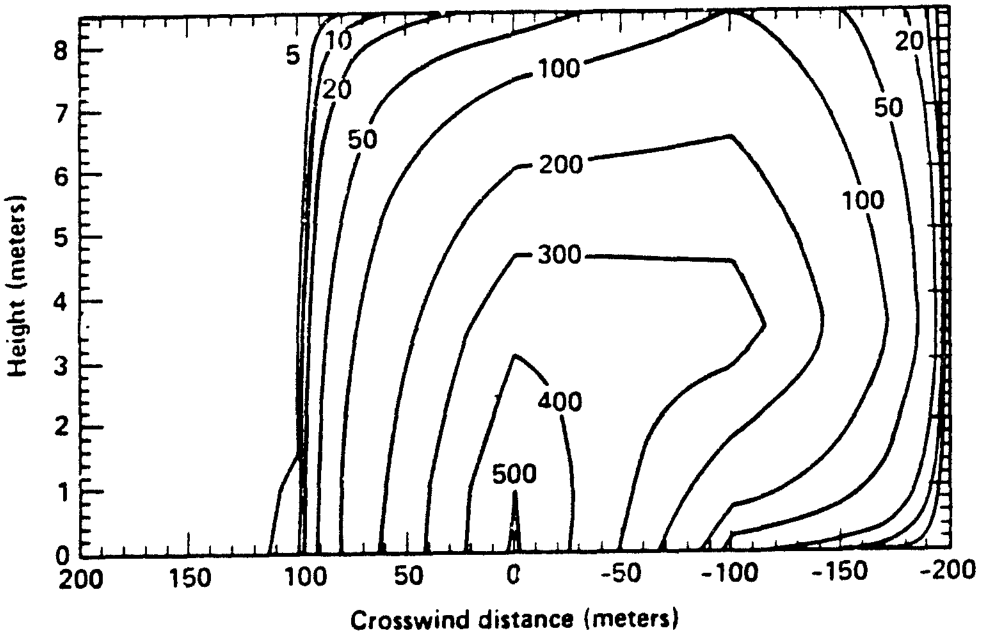


Fig. 8. Eagle 3 Crosswind Concentration Contours at 785 m.