

The emission and chemistry of reactive nitrogen species in the plume of an Athena II solid-fuel rocket motor

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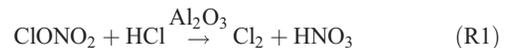
[1] In situ measurements of NO, NO_y, HNO₃, ClO, CO₂, H₂O and particles were made in the plume of an Athena II solid-fuel rocket motor (SRM) with instruments onboard the NASA WB-57F high-altitude aircraft. By normalization to CO₂, the NO_y emission index was calculated to be 2.7±0.6 g NO₂ (kg fuel)⁻¹. HNO₃ is a significant and evolving component plume NO_y. The principal source of HNO₃ is thought to be the heterogeneous reaction between ClONO₂ and HCl on emitted alumina particles. These measurements provide the first experimental evidence for the production of HNO₃ and the activation of chlorine on SRM-emitted alumina particles in the lower stratosphere. *INDEX TERMS*: 0305 Atmospheric Composition and Structure: Aerosols and particles (0345, 4801); 0322 Constituent sources and sinks; 0340 Middle atmosphere—composition and chemistry. *Citation*: Popp, P. J., et al., The emission and chemistry of reactive nitrogen species in the plume of an Athena II solid-fuel rocket motor, *Geophys. Res. Lett.*, 29(18), 1887, doi:10.1029/2002GL015197, 2002.

1. Introduction

[2] Current SRMs release energy through the oxidation of aluminum in the presence of ammonium perchlorate (NH₄ClO₄), resulting in the emission of combustion products directly into the stratosphere during launch [Prather *et al.*, 1990]. Nitric oxide (NO) is produced directly by the SRM combustion process and in secondary combustion processes known as afterburning when ambient air is entrained into the hot exhaust plume [Bennet and McDonald, 1994]. NO production and reaction in SRM plumes is not well studied. Although an SRM NO emission index of

7 g of NO (as equivalent NO₂) per kg of fuel has been reported, this value did not account for the post-emission oxidation of NO in the plume [Potter, 1977].

[3] Observations of chlorine monoxide (ClO) in a stratospheric SRM plume [Ross *et al.*, 2000] suggest that emitted NO is oxidized and subsequently forms chlorine nitrate (ClONO₂). Laboratory studies have shown that ClONO₂ could then react with hydrogen chloride (HCl) on alumina (Al₂O₃) particles via



[Molina *et al.*, 1997] to effectively convert NO to nitric acid (HNO₃). It should also be noted that (R1) provides a means of converting HCl, the primary chlorine species emitted by SRMs [Prather *et al.*, 1990], to active chlorine. The severe ozone (O₃) loss observed locally in SRM plumes is known to result from reactions involving active chlorine species [Ross *et al.*, 1997a, 1997b]. While it has been suggested that heterogeneous chlorine activation could play a role in plume O₃ loss [Ross *et al.*, 2000], the contribution of (R1) to active chlorine in the plume requires further observational support.

[4] We report here in situ measurements of total reactive nitrogen (NO_y = NO + NO₂ + ClONO₂ + 2N₂O₅ + HNO₃ . . .), NO, and HNO₃ in the stratospheric plume of an Athena II SRM. No such in situ measurements of HNO₃ and NO_y have been reported previously. These measurements are used to calculate an NO_y emission index for the SRM and to infer the occurrence of (R1) on SRM-emitted alumina particles in the lower stratosphere.

2. Results and Discussion

[5] The Athena II SRM plume was sampled following launch from Vandenberg Air Force Base, CA (34°48' N, 120°37' W) on 24 September 1999 at 66060 s Universal Time (UT). In situ measurements were conducted onboard the NASA WB-57F high-altitude research aircraft as part of the NASA Atmospheric Chemistry of Combustion Emissions Near the Tropopause (ACCENT) mission. Species measured include NO and NO_y [Weinheimer *et al.*, 1993], HNO₃ [Neuman *et al.*, 2000], ClO [Ross *et al.*, 2000], carbon dioxide (CO₂) [Gates *et al.*, 2002], water vapor (H₂O) [May, 1998], and particles [O. Schmid *et al.*, manuscript in preparation, 2002]. The WB-57F rocket plume sampling methodology has been presented in detail elsewhere [Ross *et al.*, 1997a, 1997b]. The plume was sampled a total of 6 times, with the first 5 intercepts occurring at altitudes between 18.3 and 18.5 km in the

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lower stratosphere. The final plume encounter occurred at 16.5 km during the WB-57F final descent, and is not considered in any further analysis here.

2.1. NO_y Emission Index

[6] Time series measurements obtained during the 5 plume intercepts are shown in Figure 1. Plume ages, as measured from the time of emission, ranged from 3.8 to 26.3 min. The duration of the intercepts varied from 2.2 to 12.9 s, corresponding to flight paths of 0.4 to 2.1 km, respectively. To reach these dimensions, an SRM exhaust plume must undergo near-complete dilution with ambient air. NO_y, HNO₃, CO₂, and ClO mixing ratios, however, still increased substantially above background values in the plume. CO₂ values are shown as the amount above the stratospheric background value of approximately 368 ppmv. NO values in the plume as soon as 3.8 min after emission indicate a depletion from the background value of approximately 420 pptv to 125 pptv at the time of the first intercept and to smaller amounts in the remaining intercepts. This measurement illustrates the rapid chemical conversion of NO to higher oxides of nitrogen in the plume and emphasizes the importance of reporting an NO_y emission index using a measurement of all NO_y species.

[7] In calculating an NO_y emission index, the plume dilution and expansion exhibited in Figure 1 is accounted for by normalization to a conserved species with a known emission index such as CO₂. Since the SRM emission index of CO₂ is known to be 382 g (kg fuel)⁻¹, based on the carbon content of the fuel burned, measured NO_y can be directly related to fuel use by the relative abundances of NO_y and CO₂ in the plume. Comparing the emitted quantities of NO_y and CO₂ during the plume encounters shown in Figure 1 is accomplished by integrating both species after subtracting their respective background values. The integral comparisons are meaningful because each in situ measurement is made continuously or near-continuously in a constant sample flow [Fahey *et al.*, 1995a, 1995b].

[8] The plume-integrated values of NO_y and CO₂ (ΔNO_y and ΔCO₂, respectively) during each of the 5 plume intercepts are shown in Table 1. Although NO_y and CO₂ are conserved in the plume downstream of the afterburning region, the integrated values of both species vary by more than a factor of 2 during the 5 intercepts. This variance results from both the differences in the WB-57F flight paths within the plume, and the non-uniform dispersion of the plume in the lower stratosphere [Beiting, 2000]. Knowing ΔNO_y and ΔCO₂ for each plume intercept, the NO_y emission index, EI(NO_y), can be calculated according to

$$EI(\text{NO}_y) = EI(\text{CO}_2) \cdot \frac{\Delta\text{NO}_y}{\Delta\text{CO}_2} \cdot \frac{46}{44} \text{ g NO}_2(\text{kg propellant})^{-1} \quad (1)$$

[Fahey *et al.*, 1995a] where EI(CO₂) is the SRM CO₂ emission index and (46/44) represents the molar mass ratio of NO_y (expressed as equivalent mass of NO₂) to CO₂.

[9] Using equation 1, EI(NO_y) values calculated for each of the 5 plume events yield an average of 2.7±0.6 g NO₂ (kg fuel)⁻¹ (Table 1). This value is higher than the prediction of 1.2 g NO₂ (kg fuel)⁻¹ calculated by one of the authors (P.F.Z.) using a plume flowfield model of the Athena II SRM initialized for conditions at 18.6 km altitude. For related

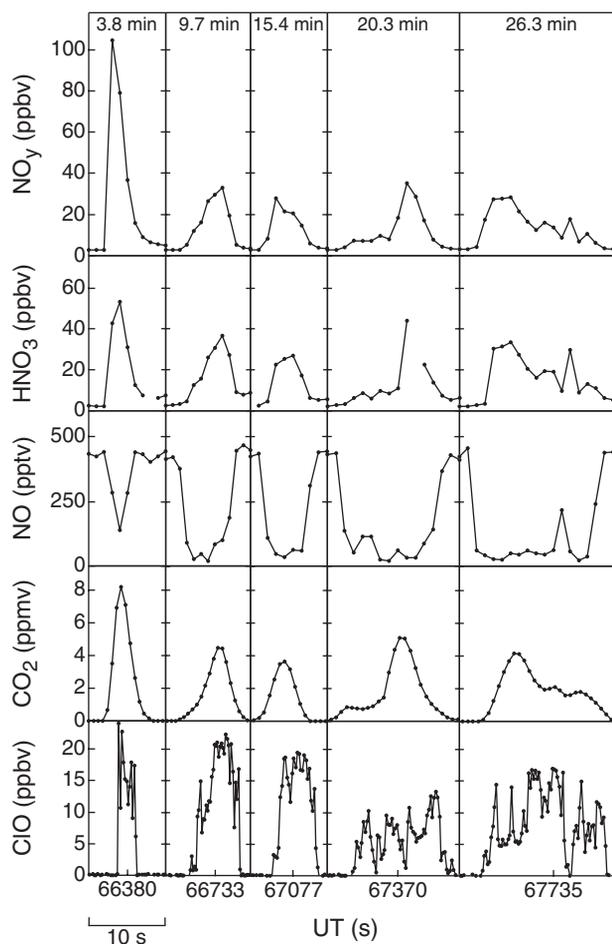


Figure 1. In situ measurements of NO_y, HNO₃, NO, CO₂, and ClO in the plume of an Athena II SRM launched from Vandenberg Air Force Base, CA on 24 September 1999 at 66060 UT. The panels showing data during the first plume intercept at 66380 UT represent 10 s of data. Panels for the remaining plume intercepts have the same horizontal scale. Plume ages at the time of intercept are shown at the top of each column.

examples of SRM plume chemistry calculations using supersonic SRM exhaust flowfield models, see Zittel [1994] and Denison *et al.* [1994]. Approximately 30% of the modeled NO production occurs in the SRM combustion chamber where the conditions affecting NO production are not well understood. The remaining NO production occurs primarily via the Zeldovich thermal mechanism [Blazowski and Sawyer, 1975] in the afterburning region of the plume and is highly sensitive to local temperature and mixing. Because of the uncertainties in afterburning plume conditions introduced by approximations to supersonic turbulent mixing, as well as the possibility that additional NO production paths may be discovered to be significant in the unusual SRM plume environment, the modeled NO production is considered an estimate and is easily subject to a factor of 2 uncertainty. While the model does not follow the reactions of NO subsequent to afterburning, the calculated NO residue can nonetheless be considered the sum of the NO_y species at all later times in the plume and is therefore directly comparable to the experimentally determined value. The comparable EI(NO_y) values from the measurements and model predic-

Table 1. Details of the WB-57F Intercepts of the Athena II SRM Plume

Plume Int.	UT (s)	Age ^a (min)	T (K)	P (hPa)	TAS ^b (m/s)	Δt^c (s)	ΔNO_y^d (ppbv·s)	ΔCO_2^d (ppmv·s)	EI(NO _y) (g NO ₂ /kg)	ΔHNO_3^d (ppbv·s)	HNO ₃ /NO _y Observed	HNO ₃ /NO _y Lower Limit
1	66380	3.8	208	70	202	2.2	178	20.3	3.5	114	0.64	0.12
2	66733	9.7	208	69	191	5.7	125	17.8	2.8	147	1.18	0.23
3	67077	15.4	207	67	173	4.2	80	11.6	2.8	93	1.16	0.22
4	67370	20.3	207	69	181	8.7	122	24.6	2.0	152	1.25	0.24
5	67735	26.3	207	66	166	12.9	192	31.6	2.4	243	1.27	0.24

^a Assumes passage of Athena II SRM through intercept altitudes at 66154 UT.

^b True air speed of the WB-57F at the time of the plume intercept.

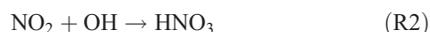
^c Elapsed time of the plume intercept, defined as the full-width at half-maximum of the CIO instrument response in the plume.

^d Δ indicates plume-integrated value with background atmospheric values subtracted. At intercept altitudes, the fuel mass flow rate of the Athena II SRM second stage was 590 kg s⁻¹, the vertical velocity was 396 m s⁻¹ and the flight path angle was 48° from vertical.

tions represent satisfactory agreement considering the uncertainties in simulating the combustion chamber and afterburning plume conditions responsible for NO production.

2.2. HNO₃ Production

[10] The relative plume-integrated values of HNO₃ (ΔHNO_3) and NO_y shown in Table 1 indicate that HNO₃ is a significant and evolving component of NO_y in the aging rocket plume. The amount of HNO₃ produced within the SRM and the afterburning plume is uncertain and is not addressed directly by the plume flowfield model. Nonetheless, any increase in HNO₃ observed during the sampling passes must be produced in situ in the plume. It is also possible that all of the HNO₃ is formed in this manner. Such production could occur in the gas phase or heterogeneously on emitted alumina particles. The most likely gas-phase production pathway,



is expected to be negligible, since it requires OH to be in excess of 1 ppbv to produce nominal HNO₃ values. Although there are no direct measurements of OH in the Athena II plume, the flowfield model predicts a negligible residue of OH in the cool plume. If the model is accurate, the OH density is too low by several orders of magnitude to explain the observed levels of HNO₃.

[11] The heterogeneous production of HNO₃ has been demonstrated in laboratory studies to occur via (R1) on the surface of alumina particles [Molina *et al.*, 1997]. NO produced directly by the SRM would be very short lived in the young (<3.8 min old) plume and efficiently converted to ClONO₂. Measurements in the plume indicate that less than 0.1% of the emitted NO remains after 3.8 min, while at the same time NO_y levels exceed 100 ppbv (Figure 1). This rapid NO removal likely occurs by reaction with ClO in the plume:



With 15 ppbv of ClO present (the approximate mean abundance of ClO in the plume during the first intercept, see Figure 1), the lifetime of NO in (R3) is approximately 1 s. The subsequent formation of ClONO₂, via



results in an NO₂ lifetime of approximately 23 s. It should be noted that the lifetimes of NO and NO₂ stated here are upper limit estimates, considering that the ClO abundance early in the plume was likely higher than the measured value of 15 ppbv at 3.8 min. The net result of (R3) and (R4) is that NO_y not already in the form of HNO₃ is rapidly sequestered

into the relatively stable reservoir species ClONO₂ in the young SRM plume. Using a reaction probability of 0.02 for (R1) [Molina *et al.*, 1997], and an observed alumina particle surface area density in the plume of $1.8 \cdot 10^3 \mu\text{m}^2 \text{cm}^{-3}$ (data not shown), the lifetime of ClONO₂ through (R1) in the young plume is 8.6 min. Thus, substantial HNO₃ can be produced via (R1) in the Athena II SRM plume by the time of the first intercept at a plume age of 3.8 min.

[12] The observed HNO₃ to NO_y ratio is 0.64 during the first plume encounter and increases above unity for the remaining intercepts (Table 1). Although the increases above unity are within the combined uncertainty of the measurements, ratios less than or equal to one are expected based on measurements made prior to the plume intercepts and during a previous flight [Neuman *et al.*, 2000]. The HNO₃ to NO_y ratios greater than unity observed here are interpreted to be the result of some fraction of the HNO₃ being adsorbed on alumina particles present in the exhaust plume. Size-resolved measurements of particles in the plume, assumed to be composed of alumina, indicate a tri-modal distribution of particle surface area (SA) in the plume. The largest particle mode, representing 65% of the surface area in the plume, consists of particles with a mean diameter of 1.7 μm . The remaining particle surface area in the plume resides in smaller modes at 0.08 μm and 0.16 μm [O. Schmid *et al.*, manuscript in preparation, 2002].

[13] The HNO₃ instrument samples through a forward-facing, sub-isokinetic inlet, which causes particles larger than approximately 0.1 μm in diameter to be significantly enhanced in the sample flow [e.g. Northway *et al.*, 2002]. In contrast, little to no particle enhancement is expected in the side-facing NO_y inlet. Thus, if HNO₃ is adsorbed on alumina particles, the observed HNO₃ mixing ratio in the plume may exceed that of NO_y when HNO₃ constitutes a significant fraction of the NO_y present. A lower limit for the HNO₃ to NO_y ratio in the plume is derived by assuming that all HNO₃ in the plume is adsorbed on alumina particles. Using a calculated enhancement function for the HNO₃ inlet [e.g. Northway *et al.*, 2002] to adjust for particle enhancement, the lower limit ratio ranges from 0.12 at a plume age of 3.8 min to 0.24 at a plume age of 26.3 min (Table 1). If any HNO₃ is in the gas phase the effect of particle enhancement will be reduced, and the HNO₃ to NO_y ratio will be greater than the lower limit values and approach the observed ratios. The actual HNO₃ to NO_y ratio can only approach unity if almost all HNO₃ in the plume is in the gas phase and the effect of particle enhancement is minimized.

[14] The increase in the HNO₃ to NO_y ratio observed in the plume between the first and second plume encounters is

likely due to continued in situ production of HNO_3 . Although there is uncertainty in the effective surface area density in the plume, the lifetime of ClONO_2 in the production of HNO_3 is long enough that (R1) could still be occurring. The near-constant HNO_3 to NO_y ratios after the second encounter are likely due to the cessation of HNO_3 production in the plume. Cessation could occur if ClONO_2 is completely converted to HNO_3 or if (R1) slows due to contamination of the alumina surface by adsorbed HNO_3 , as suggested by Molina *et al.* [1997].

[15] As a final consideration, we note that nitric acid trihydrate (NAT; $\text{HNO}_3 \cdot 3\text{H}_2\text{O}$) is not stable in the aging plume. Water vapor mixing ratios are near 20 ppmv during the first plume intercept and 11–15 ppmv for the later crossings (data not shown). For 20 ppmv H_2O and the measured plume temperature of 208 K at 70 hPa, HNO_3 mixing ratios must exceed approximately 140 ppbv for NAT to be stable [Hanson and Mauersberger, 1988]. This value far exceeds the maximum HNO_3 mixing ratio of 51 ppbv observed during the first plume intercept. As H_2O mixing ratios drop to 11–15 ppbv in the later plume crossings, NAT continues to be unstable at the observed HNO_3 mixing ratios. Before the first crossing (at a plume age of 3.8 min), NAT may have been thermodynamically stable for a brief period in the expanding plume because the plume cools to ambient temperatures in a few seconds and the HNO_3 saturation mixing ratio depends non-linearly on the H_2O mixing ratio. In addition, NAT would be present at all the plume crossings if the ambient temperatures were slightly lower. Comprehensive plume models should include the possibility of NAT formation and the associated heterogeneous reactions on NAT particles.

3. Implications

[16] The results presented here provide the first measurements of the NO_y emission index for an SRM, highlighting the importance of validating the parameterizations and assumptions used in afterburning models. The accumulation of NO_y emitted by SRMs could potentially affect O_3 production and loss processes in the stratosphere. A typical SRM launch scenario described by Danilin *et al.* [2001] results in the combustion of $3.4 \cdot 10^6$ kg of fuel in the stratosphere annually. The calculated NO_y emission index of 2.7 ± 0.6 g NO_2 (kg fuel) $^{-1}$ presented here, therefore, results in the annual emission of $9.2 \cdot 10^3$ kg of NO_y directly into the stratosphere. In contrast, the production of reactive nitrogen in the stratosphere from the decomposition of N_2O is approximately $1.5 \cdot 10^9$ kg NO_2 yr $^{-1}$ [Murphy and Fahey, 1994]. Thus, the contribution of SRM emissions to the stratospheric NO_y budget is negligible compared to that from natural sources. Future NO_y emissions from other rocket propellant types (i.e. hydrazine-based fuels) may not be negligible.

[17] These data also provide the first experimental evidence for the production of HNO_3 via (R1) on SRM-emitted alumina particles in the lower stratosphere. In addition, they provide evidence that HNO_3 may remain adsorbed on the alumina particles. Although the occurrence of (R1) results in chlorine activation in the plume, the importance for local O_3 loss is not clear since active chlorine is otherwise abundant in the plume [Ross *et al.*, 1997a, 1997b]. With the potential future increase in launch vehicle traffic, understanding how reactions on alumina particles affect global stratospheric O_3 will be critical in assessing the effects of the space launch industry.

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