Evaluation and Use of Stand-Alone Commercial Photolytic Converters for Conversion of NO$_2$ to NO

Keith G. Kronmiller

ManTech Environmental Technology, Inc., P.O. Box 12313, Research Triangle Park, NC 27709

William A. McClenny

National Exposure Research Laboratory, U.S. Environmental Protection Agency, Research Triangle Park, NC 27711

ABSTRACT

Two types of stand-alone photolytic converters of nitrogen dioxide (NO$_2$) to nitric oxide (NO) are now commercially available for use with NO, ozone (O$_3$) chemiluminescence detector (CLD) monitors for the measurement of NO$_2$. Both units have been tested for interferences resulting from photolysis of nitrous acid (HONO) and from the decomposition of peroxyl acetyl nitrate (PAN). One unit (Model 81800, Spectra-Physics Stratford, CT) is based on the use of a broadband source (short-arc mercury lamp) and incorporates a source cooler. This unit has been used for two month-long field studies, one in May 2002 and a second in October 2003. The results indicate that the converter is robust and reliable with conversion efficiencies (CEs) of 35–70% depending on the airflow rate through the converter. The second commercial unit (Droplet Measurement Technologies–Sonoma Technology, Inc., Petaluma, CA) is based on a light-emitting diode (LED) array with output emission wavelengths centered near 390 nm with emission bandwidths of typically 20 nm. This unit is being field tested for use as part of ongoing tests. Based on results so far, the prospect of using one of the stand-alone converters with an external, heated metal (molybdenum) converter and a chemiluminescence monitor to measure NO, NO$_2$, and NO$_Y$ (e.g., NO, NO$_2$, HNO$_3$, HONO, HO$_2$NO$_2$, NO$_3$, N$_2$O$_5$, and organic nitrate) seems reasonable.

INTRODUCTION

Commercial chemiluminescence analyzers can easily provide NO measurements with accuracies well under 0.2 ppb.[1] Other reactive nitrogen-containing gases are converted to NO (by a heated metal, for example) so that, if the conversion is complete, the sum of NO and the other gases is equal to total reactive nitrogen. Methods for NO$_2$ measurements using chemiluminescence analyzers rely on the fact that NO and NO$_2$ are primary emissions so that in cases where they constitute a health risk, NO$_X$ (the sum of NO and NO$_2$) is dominant over other nitrogen-containing compounds. Subtracting NO from NO$_Y$ then gives an upper limit of NO$_2$, and this limit has been used as the main equivalent method for NO$_2$ monitoring to establish compliance with U.S. ambient air quality standards. However, in modeling the atmosphere, the real concentration of NO$_2$ must be distinguished from NO$_Y$ minus NO to properly account for atmospheric chemistry. One option is photolytic conversion of NO$_2$ so ambient concentrations

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can be determined by subtraction of NO (no converter) from NO plus [CE × NO₂], and then by dividing the CE, where CE is the conversion efficiency established with NO_2 calibration gas. Ideally, these two concentrations are established at the same time so that the subtraction can be exact. However, in most commercial instruments, the two concentrations are measured sequentially by passing through or bypassing the converter. As a result, high variability in ambient concentrations can introduce errors in subtraction (one concentration changes while the other is being measured). The magnitude of these errors is moderated by using frequent sequencing and averaging over adjacent cycles. Many research scientists[2,3,4] have assembled photolytic converters for use with chemiluminescence monitors, and at least one commercial supplier incorporates a photolytic converter as part of a NO and NO_2 monitoring system. Apparently, however, no separate photolytic system has been available. Recently, two stand-alone systems have been placed on the commercial market for use with chemiluminescence monitors. One is designed along guidelines provided by Ryerson[2] and uses a broadband light source. The second is designed for use with light-emitting diodes (LEDs) that can be composition tuned to emit radiation over a narrow wavelength interval in the 350–400-nm range where NO_2 is efficiently photolyzed.

Desirable characteristics of a photolytic converter include a number of interrelated merit parameters: (1) high converter efficiency; (2) low residence time in the converter (to minimize the NO, ozone (O_3) reaction); (3) low photolytic interference equivalence for other compounds, e.g., nitrous acid (HONO); (4) low-temperature operation to minimize conversion of thermally labile compounds like peroxyl acetyl nitrate (PAN); (5) ease of operation and maintenance; (6) robustness; and (7) low cost. This paper examines most of these parameters. Operation of the chemiluminescence monitor at the manufacturer's specifications for flow rate determines residence time in the photolysis chamber and the converter efficiency. Since the NO, O_3 chemiluminescence monitor measures the rate of arrival of NO molecules, high flow rates typically increase detection sensitivity while at the same time reducing the efficiency of photolysis.

Alternative analytical systems that accurately measure NO_2 have been developed. Methods such as the tunable diode laser (TDL),[5] laser-induced fluorescence (LIF),[6] differential optical absorption spectrometry (DOAS),[7] and direct luminol chemiluminescence[8] are all viable techniques that do not require conversion of NO_2. However, these methods are not currently as prevalent in the U.S. installed instrument base as the chemiluminescence-based systems. They are, however, often used for producing high-quality ambient measurements of NO_2 during intensive field research programs.

As reported here, two of the Spectra-Physics photolytic converters (Thermo/Oriel Model 81800, Stratford, CT) were used in the Bay Regional Atmospheric Chemistry Experiment (BRACE) in May 2002 in Tampa, FL, for one month, and one was used again in the second BRACE in October 2003. Atmospheric Research Associates (ARA, Plano, TX) used this type of converter in the Southeastern Aerosol Research and Characterization Study (SEARCH) at eight sites for over 18 months. Another group, the Lake Michigan Air Directors Consortium and the Illinois State Water Survey (Champaign, IL) has also used the Thermo/Oriel photolytic converter for

routine measurements. Although newer to the commercial market, the LED-based photolytic converter has been used recently onboard aircraft as reported by Buhr.[9]

EXPERIMENTAL METHODS

General Procedures

A diagram of the laboratory test apparatus used during the interference tests is shown in Figure 1. Test atmospheres of NO in air were produced by dilution of National Institute of Standards and Technology (NIST)-traceable NO compressed gas standards (Scott-Marrin, Inc., Riverside, CA). NO free air (zero air) for dilution was produced using compressed air followed by conversion of any ambient NO to NO_2 by ozonation. Activated charcoal was then used to remove any trace NO_2 and O_3 from the air. A Model TEI 146C calibration/dilution system (Thermo Environmental Instruments, Franklin, MA) was used for dilution of the NO standard gas using internal mass flow controllers and was calibrated with a BIOS Model DC-2M (BIOS International, Butler, NJ) NIST-traceable flowmeter.

NO_2 test atmospheres were produced by titrating a portion of the generated NO concentration with O_3 using the Model TEI 146C internal ozone generator. A Model 49PS NIST-traceable O_3 analyzer was used to verify the O_3 used for titrating the NO.

HONO was produced by reverse permeation of hydrochloric acid (HCl) vapor carried by N_2 into sodium nitrite ($NaNO_2$) as described by Febo et al.[10] Dilution of the generated HONO vapor took place outside the multi-port sampling manifold using zero air from the Model TEI 146C. Prior to experiments, adequate time was allowed for conditioning the system components, which consisted of 1/8- and 1/4-inch Teflon tubing and fittings as well as the multi-port glass sampling manifold. The upper limit of HONO concentration was determined using a second chemiluminescence analyzer operated with a heated metal converter (moly).

HNO_3 was produced using a permeation tube (VICI Metronics, Santa Clara, CA) held inside a glass chamber within an insulated enclosure. The chamber temperature was regulated at 50 °C. Selection for either HONO or HNO_3 was facilitated using Teflon solenoid valves, which could be activated manually with a switch. The upper limit of HNO_3 concentration was determined with a chemiluminescence analyzer in the same way as for HONO.

PAN was prepared for exposure experiments by nitration of tridecane as described by Gaffney et al.[11] Two methods were used to deliver PAN at low concentrations to the sampling manifold. In the first, an 80-L Teflon bag was flushed with nitrogen, then an aliquot of the liquified PAN in tridecane was injected into the bag, followed by refilling with nitrogen. The filled bag was then transferred to the experimental apparatus laboratory for testing. In the second method, the liquified PAN in tridecane was placed into a midget impinger. Zero air was used to carry the evaporated PAN from the headspace above the liquid to a separate dilution vial. Additional zero air was then used to reduce the PAN concentration. Using this method, a clean 80-L Teflon bag was filled from the common manifold for analysis with a GC/ECD system for comparison purposes. The upper limit of PAN was estimated using a chemiluminescence analyzer in the same way as for HONO and HNO_3.

Figure 1: Apparatus Used during Interference Evaluation Study
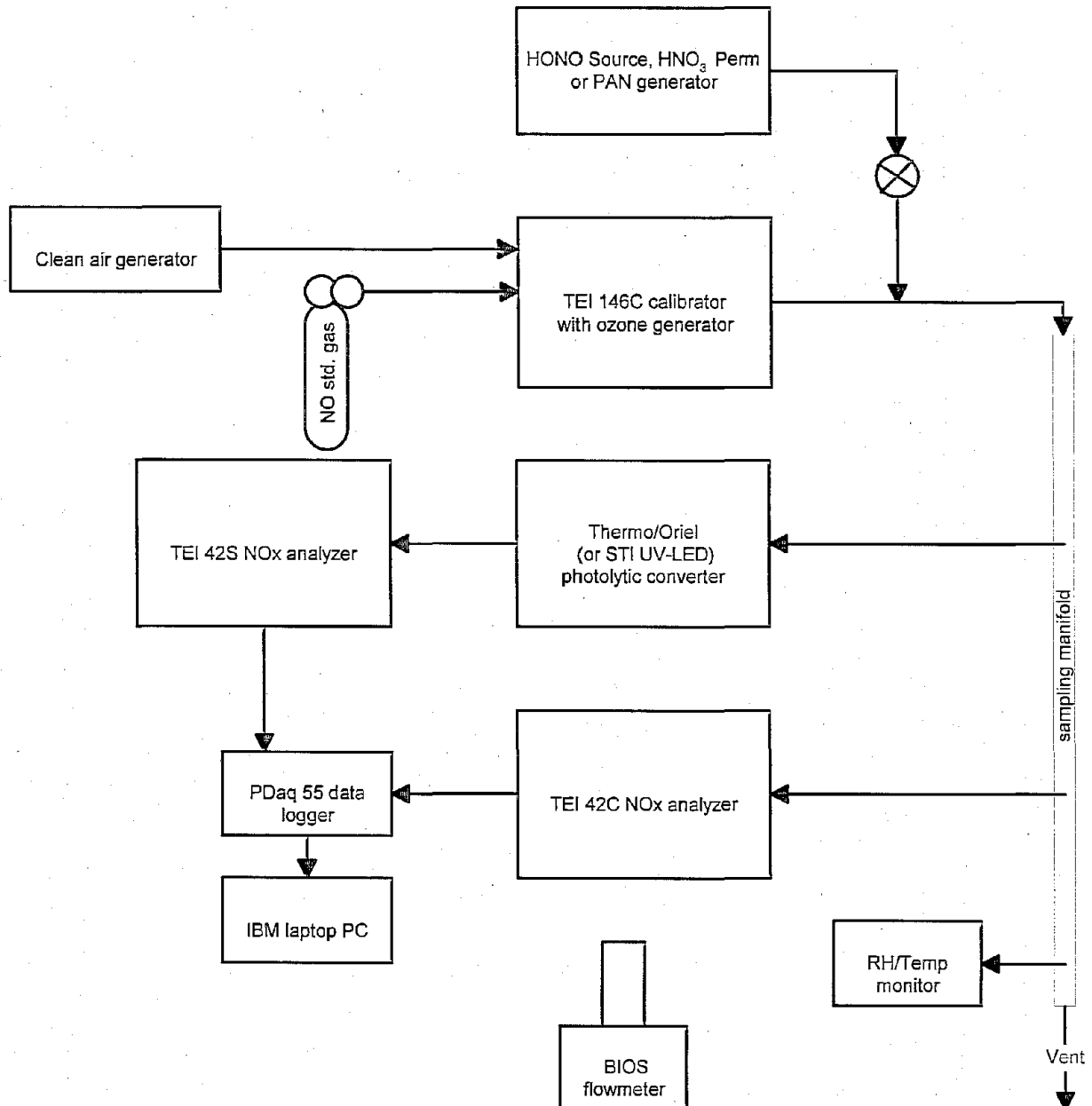
Broadband Photolytic Converter

Conversion Stability

Laboratory testing of the Spectra-Physics Model 81800 NO_2 photolytic converter using the calibration system shown in Figure 1 demonstrated that CE can vary a few percent. Changes are most likely caused by degradation of the source lamp inside the device. Changes in the CE directly affect the outcome of the mathematical calculation of the NO_2 concentration. The accuracy of the CE parameter is more important at lower CE values as it has a greater effect on the calculated NO_2 concentration.

To maintain measurement accuracy within acceptable quality assurance guidelines, calibration and CE determination should be performed daily. Under constant conditions of light intensity and residence time in the photolytic cell, the broadband light source (and CE) was observed to decrease gradually over time with small intensity variations around the trend line. Frequent calibrations and NO_2 CE determinations allow the data acquired between two consecutive cycles to be adjusted using standard interpolation methods. The Spectra-Physics Model 81800 NO_2 converter was used during two field monitoring campaigns of the Bay Regional Atmospheric Chemistry Experiment (BRACE) in May of 2002 and September–October 2003. In the 2002 experiment, two sites were operated with identical sets of instruments and calibration apparatus. During the 2003 study, measurements were taken only at one site. Zero, span, and CE determinations were performed daily and the results of these show the converter has sufficient CE stability for high-quality NO_2 data to be reported.

Table 1 summarizes the CEs determined at the field sites and the analyzers' span response over the study periods. The data show that from one day to the next the CE stability is less than 1%.

Table 1. Average CE and span stability of the stand-alone NO_2 photolytic converter and the chemiluminescence analyzers used during two month-long field monitoring studies.

Site	Photolytic CE	Average Daily Change in CE	NO Channel Span	Photolytic Channel Span
Gandy 2002[a]	36.7% ± 0.9	-0.19%	0.998 ± 0.07	1.000 ± 0.02
Sydney 2002[a]	41.9% ± 2.1	-0.5%	1.001 ± 0.03	1.002 ± 0.02
Sydney 2003[b]	80.2% ± 4.2	-0.45%	1.016 ± 0.04	1.007 ± 0.02

[a]TEI 42CTL flow rate 1300 cm³/min.
[b]Environnement S.A. AC31M flow rate 350 cm³/min.

Interference of Other Species

The specificity of the photolytic conversion of NO_2 by broadband light in the wavelength region of 350–400 nm has been investigated by others[2] and is a function of absorption cross sections of species other than NO_2 likely to occur in the environment. HONO is a major potential interferent, although its absorption cross section is significantly lower over the wavelength range produced by the photolytic light source.[2] Laboratory tests performed using the HONO generation system supported these assumptions. The data shown in Table 2 summarize the results.

Table 2. HONO interference test data for the Spectra-Physics Model 81800 photolytic converter. HONO CE avg. = 10.4% ± 2.0%.

Date	HONO Concentration ppb	Light On ppb	Light Off ppb	NO$_2$ Conversion Efficiency %	HONO Conversion Efficiency %
10/08/02	15.7	1.49	0.100	42.0	8.8
10/09/02	15.5	1.47	0.300	42.0	7.5
10/31/02	11.7	1.33	0.060	42.1	10.8
10/31/02	18.0	1.82	0.060	42.1	9.8
11/04/02	20.2	2.12	0.040	42.3	10.3
11/05/02	16.1	1.71	0.026	42.3	10.5
11/05/02	16.1	1.69	0.036	42.3	10.3
01/03/03	30.2	2.38	0.020	37.7	7.9
01/09/03*	20.3	2.99	0.010	52.2	14.7
01/22/03	16.8	1.98	0.050	49.0	11.5
01/23/03	16.6	2.06	0.026	49.0	12.3

*Oriel lamp changed 01/09/03.

PAN Interference Test

Other species that potentially cause inaccuracies in measurement of NO$_2$ by photolytic conversion may not have absorption cross sections within the photolytic lamp spectrum, but may decompose into NO$_2$ and thus undergo photolysis. PAN thermally decomposes during transient time through the analytical system components because of the elevated temperatures.[12] Tests were performed to evaluate the extent of the decomposition and its effect on NO$_2$ measurements (see Table 3). Various dilution flows were established with the dynamic dilution system to provide a range of PAN concentrations. Changes in the chemiluminescence detector (CLD) NO channel were recorded with and without the photolytic light. The increased NO readings were then divided by the PAN concentration (measured by the heated molybdenum [moly] converter NO$_Y$ channel).

Table 3. PAN interference test data.

PAN Concentration ppb	Light On ppb	Light Off ppb	NO$_2$ Conversion Efficiency %	PAN Conversion Efficiency %
15.3	0.290	0.034	44	1.7
14.0	0.278	0.027	44	1.8
3.0	0.161	0.051	44	3.4
3.2	0.160	0.040	44	3.7

Decomposition of the PAN in the system plumbing prior to delivery into the photolytic chamber was tested by filling an 80-L Teflon bag followed by analysis using a pulse-discharge electron capture detection (ECD) monitor developed for real-time monitoring.[13] Analysis of the PAN collected during simultaneous NO_Y measurements showed very little decomposition prior to delivery into the photolytic system. If PAN decomposed to NO_2 prior to analysis and collection, the PAN gas chromatography (GC) with ECD system would report lower values. The GC-ECD analysis agreed within 5% of the NO_Y measurements, so there was little decomposition inside the system before the photolytic converter. The 2–4% CE observed was then thought to be due to decomposition inside the photolytic chamber caused by the elevated temperature (from the high-energy UV lamp and heat produced by the internal power supplies).

Solid-State UV-LED Photolytic Converter

Conversion Stability

The Droplet Measurement Technologies–Sonoma Technology, Inc. (STI, Petaluma, CA) UV-LED photolytic converter, although packaged in a much smaller form than the Spectra-Physics broadband light converter, was found to have only half the CE for NO_2. As discussed earlier, variability at low CEs will result in larger uncertainties in the reported NO_2 concentrations. One factor that contributes to the CE is residence time within the photolytic chamber. Slower sample flow rates and longer residence times increase the CE, as shown in Table 4.

Table 4. STI UV-LED photolytic conversion efficiency with analyzers from various manufacturers showing effect of residence time.

CLD Analyzer	Flow Rate cm³/min	Residence Time sec	Conversion Efficiency %
TEI Model 42S	1440	0.625	21.1 ± 0.20
TEI Model 42S	1050	0.857	26.6 ± 0.14
TEI Model 42S	1030	0.874	29.0 ± 0.66
TEI Model 42CTL	1200	0.750	27.4 ± 0.44
Environnement S.A. Model AC31M	750	1.200	43.7 ± 1.50

Interference of Other Species

Although the radiant ultraviolet (UV) light intensity from the solid-state diode array used in the STI converter is much less than that provided by the mercury (Hg) lamp used in the Spectra-Physics converter, it is confined to a narrower band of wavelengths. This provides less interference due to photolytic conversion of HONO. This is shown in Table 5.

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Table 5. TEI 42S with STI UV-LED in-line with sample inlet (flow rate = 1030 cm³/min, average % HONO CE = 1.74% ± 0.22).

Date	HONO Concentration ppb	Light On ppb	Light Off ppb	NO₂ Conversion Efficiency %	HONO Conversion Efficiency %
01/22/04	7.0	0.15	0.046	29.8	1.5
01/22/04	7.1	0.16	0.020	29.6	1.9
01/23/04	7.2	0.15	0.016	29.6	1.9
01/23/04	6.9	0.18	0.040	29.6	1.9
01/28/04	6.8	0.16	0.060	29.8	1.5

HNO_3 Interference Test

Based on the lack of significant absorption characteristics close to the available wavelengths produced by the solid-state UV-LED converter, we did not expect HNO_3 to be converted. A test conducted using the Environnement S.A. Model AC31M with the STI UV-LED in place of the internal moly converter showed this is the case. With a HNO_3 concentration of 19 ppb, the apparent HNO_3 CE was found to be 1.1%.

PAN Interference Tests

The solid-state UV-LED-based STI converter operates at a much lower temperature than the Spectra-Physics photolytic converter. As a result less thermal dissociation of the PAN into NO_2 was expected. The dynamic dilution of PAN headspace method was used with simultaneous collection of a sample bag for GC-ECD analysis. Results are shown in Table 6.

Table 6. PAN interference tests using the STI UV-LED photolytic converter with TEI 42S (flow rate = 1030 cm³/min), using dynamic PAN dilution apparatus with Teflon bag fill during measurement. PAN GC-ECD analysis agreed within 5%.

Date	PAN Concentration ppb	Light On ppb	Light Off ppb	NO₂ Conversion Efficiency %	PAN Conversion Efficiency %
01/29/04	252.00	0.880	0.410	29.8	0.2
01/29/04	8.82	0.048	0.032	29.1	0.2
01/30/04	3.67	0.022	0.016	28.9	0.2

These results confirm there was very little decomposition in the system either during delivery to the converter or inside the STI UV-LED converter.

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FIELD MEASUREMENTS

Broadband Photolytic Converter

Tampa BRACE Study, May 1–31, 2002

U.S. Environmental Protection Agency (EPA) scientists and ManTech personnel measured NO and NO_2 data taken at two separate locations during the month-long study around the Tampa Bay area. One site (Gandy) was located at a Hillsborough County Environmental Protection Commission air pollution monitoring station near the east end of the Gandy Bridge spanning Tampa Bay. The second site (Sydney) was located 35 km east-northeast of the Gandy site on land belonging to the county water department, south of Sydney Road and east of Valrico Road in Brandon, FL.

One of the study participants, ARA, recorded total nitrogen compounds as NO_Y and also provided nitric acid (HNO_3) by denuder difference in real time. Although both the ARA and the EPA/ManTech data were from point monitoring analyzers, the inlet sampling level for the ARA instrument was approximately 5 m above the EPA inlet. Thus, a low correlation is possible due to spatial variations in atmospheric concentrations between the two different sampling probe heights. ARA provided data from both the Sydney and Gandy sites for the entire study. Their data, $[NO_Y - HNO_3]$, can be considered the upper limit of $[NO_X]$, which, if different from the sum of [NO] and $[NO_2]$, should be due to other nitrogen-containing compounds (e.g., HONO, HO_2NO_2, NO_3, N_2O_5, organic nitrate, and particulate nitrates).

Sydney Site ARA NO_Y* ($NO_Y - HNO_3$) versus EPA/ManTech NO_X ($NO + NO_2$)

Comparing the ARA NO_Y* data set with the EPA photolytic NO_X set required producing a combined data set with matching time intervals and with only paired data. After this process, hourly averages were produced, leading to the data charted in Figures 2 and 3. Interestingly, the data show the EPA photolytic NO_X is lower during the afternoon hours than the ARA NO_Y*. Hourly averages are charted in Figure 2 showing the daily trends. Both data sets track very well (correlation $r^2 = 0.968$), especially during the evening hours when NO_2 dominates the total NO_Y. During the afternoon when there is less NO_2, the other nitrogen-containing compounds exhibit a larger influence and the results obtained with the two instruments separate more.

Figure 2: ARA Hourly Averaged Data versus EPA/ManTech NO_x, May 2002 BRACE Study
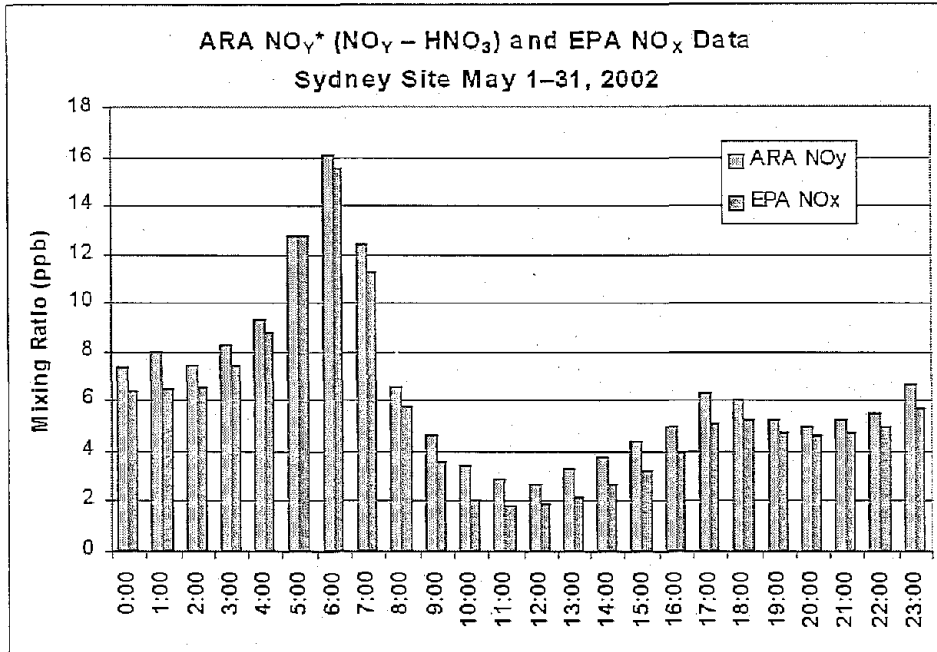
Figure 3: ARA NO_Y* (NO_Y − HNO_3) compared to EPA/ManTech NO_x (NO + photolytic NO_2)

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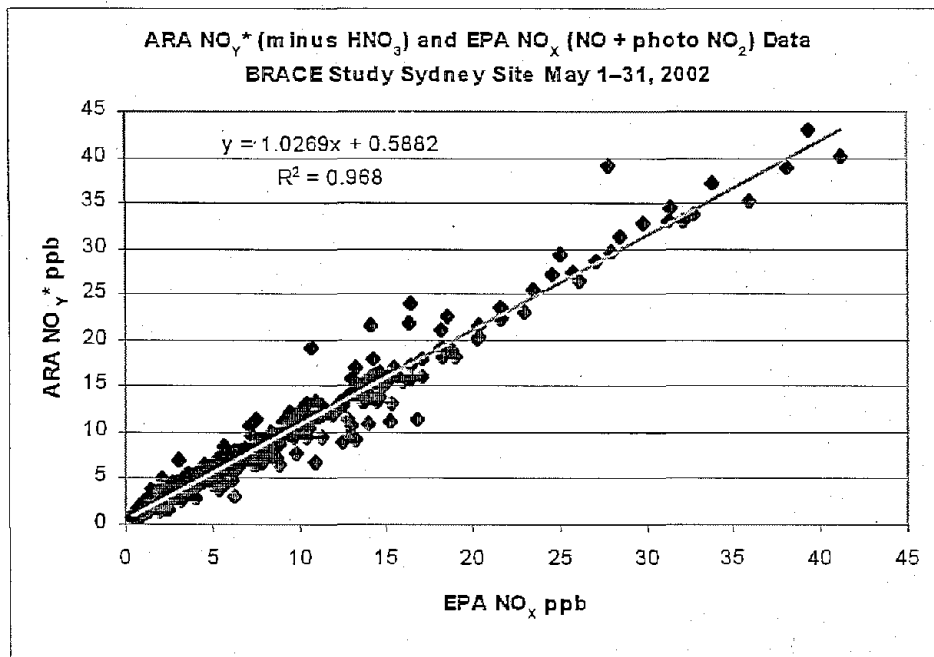
SUMMARY

Two stand-alone photolytic NO_2 converters, available as commercial products, have been evaluated. One, based on the use of a broadband UV source from a Hg lamp, is designed to be used with either research-grade or commercially available O_3 chemiluminescence analyzers. This device was found to be reliable and well engineered. Operation for long periods without operator adjustments or replenishment of the internally contained coolant was observed during two month-long intensive field studies in Tampa, FL (BRACE 2002 and BRACE 2003). Experience shows that because of the rate of change of the UV light due to lamp degradation, although low (~ -0.4%/day), users should perform a daily zero and NO_2 span check so the reported data can be of the highest quality possible. Interferences due to photodissociation of HONO inside the system have been reported.[2] Our evaluations showed this CE can be as high as15%. Optical filtering may provide a means of limiting this interference, but during these evaluations no filters were available. Equivalent PAN CE was as much as 4%, apparently due to thermal conversion of PAN to NO_2 in the instrument and converter.

The second stand-alone photolytic converter evaluated (Droplet Measurement Technologies–STI UV-LED) was also found to be reliable and well engineered. This converter is designed and packaged in a size that allows it to be installed inside a conventional chemiluminescence analyzer in place of its internal moly converter. At the time of this work, we have not had the opportunity of using this converter outside the laboratory. The CE for this system has been tested with three chemiluminescence analyzers with various sample flow rates. This system was found to have 40–50% less CE than the broadband system at identical flow rates, although reducing the inlet flow rate to 750 cm^3/min gives a CE > 40%. Although the CE is much lower than that of the broadband light system, degradation of the light intensity is less a concern because of the nature of the solid-state UV light-emitting diodes used in this device. HONO interference tests, using the more narrow wavelength spectrum that falls outside HONO absorption cross-section features, confirmed that CE of HONO is less than the broadband lamp converter (2% vs. 15%). PAN and HNO_3 were each found to have negligible interference, so these are not considered to be a problem in ambient measurements of NO_2.

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16. Abstract

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