

EPA REVIEW OF SHELL BENZENE MONITORING

Publication 1019.1*

March 2006

SUMMARY

Benzene is an aromatic hydrocarbon found in petrol and is used for a range of industrial purposes. In Corio, the Shell refinery and motor vehicles are the main sources of benzene emissions to air.

EPA Victoria has been measuring the levels of benzene in air at Geelong Grammar (north-east site) in Corio since December 2002 and at Corio, Rosewell and North Shore Primary Schools since August 2004. Average benzene concentrations in Corio were found to be similar to levels in Melbourne and lower than levels found beside busy roads (EPA publication 999, *Benzene monitoring in Corio, 2003-05*). Results indicate that benzene levels in Corio and elsewhere were below the health-based national investigation level.

Shell's EPA licence was amended in November 2004 to require it to continuously measure benzene levels at two locations in Corio. EPA has reviewed this monitoring and found issues that the company needs to address.

Shell has been measuring benzene using a monitoring device based on the CEREX instrument. A third party operates the instrument.

This report presents findings of EPA's investigation of Shell's ambient air monitoring for benzene. EPA's assessment included:

- a review of the methodology and procedures used by the operators of Shell's benzene monitoring system
- a review of the data generated between January and June 2005 at the north-west site
- comparison of data generated from a side-by-side study with EPA's instrument at the north-east site in September 2005.

The findings of the review indicate that there is significant doubt in the accuracy of benzene data being generated by Shell. The reasons for this are:

- the operators of Shell's benzene monitoring system did not have adequate systems and procedures to ensure the quality of data being produced
- the majority of the monitoring data generated during the time assessed was not reliable.

A detailed review of the monitoring data recorded by Shell's benzene monitoring system, located at the north-west site during March and April 2005, indicates that high levels of benzene were not detected.

To address these issues Shell has been directed to immediately seek National Association of Testing

* This replaces Publication 1019, issued December 2005.

EPA REVIEW OF SHELL BENZENE MONITORING

Authorities (NATA) accreditation for monitoring benzene as per the conditions of its licence.

In addition, to ensure that reliable monitoring for benzene is conducted, Shell has been directed by EPA to implement a substitute monitoring program for benzene by using a NATA-accredited method until deficiencies in the operation of Shell's benzene monitoring system are addressed.

The findings of this review do not change EPA's conclusions that benzene levels in Corio are below national investigation levels and generally similar to those in Melbourne.

EPA requires Shell to continue to reduce benzene emissions from the refinery. Benzene emissions from motor vehicles are expected to drop in 2006 due to the introduction of national cleaner fuel standards.

INTRODUCTION

Shell received a revised EPA licence in November 2004 with conditions that require improved environmental performance and monitoring of environmental pollutants.

The licence requires Shell to undertake continuous NATA-accredited benzene monitoring in two locations in Corio (Licence Condition 3.4 and Licence Condition 5.2). The locations were chosen based on predominant wind directions and to enable assessment of any potential effect on neighbouring communities.

This monitoring is required by EPA to enable Shell to gather information about benzene levels in the air and provide this information to the community.

Shell installed their first system near the corner of Princes Highway and School Road (north-west site)

in Corio in late 2004 and a second system near Geelong Grammar School (north-east site) in mid-2005.

An initial review by EPA of the quality of the data produced by Shell's benzene monitoring system at the north-west site received in mid-2005, as well as the delays in gaining NATA accreditation, prompted a more detailed investigation by EPA.

To assist all involved parties in the interpretation of the data provided, EPA (with assistance from an independent expert) conducted a review of Shell's benzene monitoring system. This included a review of the operation of the system as well as the data produced in 2005 (January to June) at the north-west site and a side-by-side operation of EPA's and Shell's systems in September at the north-east site to check data.

Both monitoring systems – OPSIS used by EPA and CEREX by Shell – are based on UV-visible spectrometers and use the technique of differential optical absorption spectrometry (DOAS). This involves passing a light beam over a specified distance to a receiver. The amount of absorption of light at a specific wavelength is proportional to the concentration of benzene or other chemicals for which these systems are calibrated.

EPA has been measuring the levels of benzene in air continuously at the north-east site in Corio since December 2002, and for one day each week at Corio, Rosewall and North Shore Primary Schools and Geelong Grammar from August 2004 to August 2005 with a canister system. Benzene levels measured in Corio were found to be similar to levels in Melbourne and lower than levels next to busy roads. Results indicate that benzene levels in Corio

EPA REVIEW OF SHELL BENZENE MONITORING

were below the health-based national investigation level (EPA Publication 999, *Benzene monitoring in Corio, 2003–2005*).

PROCEDURES REVIEW

EPA conducted a review of the methodology and procedures used by the operators of Shell's benzene monitoring system. The review involved an initial meeting with representatives of both the contractors running the equipment and Shell on 30 August 2005. At this meeting operational and quality assurance procedures were discussed.

Following the meeting, more detailed examination of the following documents was conducted:

- CEREX Environmental Services, Inc. *UV Sentry Open-path Air Monitor End-User Manual* Version 20050218-Draft
- instrument operation procedure
- daily quality assurance logs
- field air quality data records from March to July 2005
- operational logs
- The CEREX Ultraviolet (UV) Differential Optical Absorption Spectrometer CEREX Environmental Services, Inc. document *(DOAS) Statistical Interpretation of Data*, used for constructing absorption spectra and comparing spectra to reference spectra.

EPA found that the monitoring done for Shell did not have adequate systems and procedures to ensure an adequate quality of data being produced. Examples of identified deficiencies included:

- lack of a quality system to support the monitoring of benzene in ambient air
- the operators of Shell's benzene monitoring system had not verified or validated their method for monitoring benzene in ambient air
- there was no documented evidence showing regular calibration using certified and traceable gas standards
- documented procedures for post data analysis and data validations were not evident.

DATA REVIEW

A review was conducted of monitoring data generated by Shell's benzene monitoring system located north-west of the refinery between January and June 2005.

From February through to the end of June benzene levels recorded by Shell's benzene monitoring system fluctuated between positive readings and negative readings. Analysis of five-minute data showed significantly negative results on many occasions. These negative fluctuations indicate that, during this period, the instrument was not operating effectively.

EPA and an independent reviewer conducted a detailed examination of the March and April data. Five areas were examined:

1. statistical confidence parameters used to confirm the presence of benzene
2. comparison of recorded and reference benzene ultraviolet spectra
3. prevailing environmental conditions

EPA REVIEW OF SHELL BENZENE MONITORING

4. nearby benzene measurements taken by EPA at the same time
5. benzene–toluene ratios.

EPA’s review of each of these five areas shows that the benzene levels measured by Shell during March and April were not valid. Each of these is discussed below.

1. Statistical confidence parameters

To assist in confirming the presence of benzene Shell’s benzene monitoring system compares the measured ultraviolet spectrum with a reference benzene spectrum. If benzene is present then the measured and reference spectra should be the same.

The comparison is done mathematically and two statistical parameters, per cent standard error and square of correlation coefficient (R^2) are calculated. A per cent standard error approaching zero and an R^2 approaching 1 indicate benzene is present. The guide recommended by the CEREX manufacturer for interpreting R^2 values is:

R² value	Confidence
0.75–1.00	Benzene is present
0.50–0.75	Benzene presence is very likely
0.20–0.50	Benzene presence is unlikely
0.00–0.20	Benzene is not present

In March and April a total of 297 hours of data with high levels of benzene were recorded by the operators of Shell’s benzene monitoring system.

No. of hours	R² value
0	0.75–1.00
15	0.50–0.75
124	0.20–0.50
158	0.00–0.20

Based on the R^2 values, 158 of the 297 hours of data fell into the category ‘Benzene is not present’, and 124 of the 297 hours of data fell into the category ‘Benzene presence is unlikely’. This indicates that for a substantial proportion of this period Shell’s benzene monitoring system was logging benzene when benzene was either not present or unlikely to be present in the air.

2. Comparison of benzene spectra

A visual comparison of the measured and reference spectra for the 15 hours of data where the presence of benzene was very likely indicated that they were not similar. A detailed analysis of the measured spectra by an independent reviewer indicated that oxygen was causing a significant interference with the measurement of benzene.

3. Prevailing environmental conditions

Benzene levels would be more likely to rise when the wind is blowing from either Shell Refinery or the Princes Highway. Benzene levels versus wind direction are shown in Figure 1.

During the 297 hours that Shell’s benzene monitoring system gathered data in March/April, winds blew in a southerly direction (from the direction of the refinery) for 33 hours. However for approximately 264 hours Shell’s benzene monitoring system logged apparent high levels of

EPA REVIEW OF SHELL BENZENE MONITORING

benzene, when the wind was coming from a wide range of directions.

4. Nearby EPA measurements

EPA utilised two different methods to monitor benzene levels in Corio, the OPSIS system – which is similar to Shell’s benzene monitoring system – and the canister system.

Results indicated that benzene levels in Corio were below levels of concern (EPA Publication 999, *Benzene monitoring in Corio, 2003–05*).

EPA’s monitoring system located at the north-east site, a similar distance from the refinery, did not detect these high levels of benzene during the same period (Figure 2).

5. Benzene–toluene ratios

Petrol contains a number of different hydrocarbons, including benzene and toluene. There is more toluene than benzene in petrol. In the majority of cases benzene levels should be lower than toluene levels. The table below shows the benzene–toluene ratios measured by EPA at Geelong Grammar School and Shell in April 2005 when benzene levels were greater than 5 µg/m³. EPA results show higher toluene levels than benzene for the majority (76 per cent) of the time, as expected. Shell recorded lower levels of toluene than benzene for the majority (86 per cent) of the time.

Benzene–toluene ratio	EPA (per cent)	Shell (per cent)
Less than 1	76	14
Between 1 and 2	13	13
Between 2 and 10	9	40
Greater than 10	2	33

SIDE-BY-SIDE COMPARISON

During September 2005, a Shell benzene monitoring system and an EPA system were located next to each other at the north-east site. The aim of this study was to determine if the two systems measured the same levels of benzene, and to assess the Shell data in comparison with EPA data.

Hourly benzene levels measured by Shell and EPA are compared in Figure 3. It can be clearly seen that there is little correlation (note that the uncertainty of EPA data is ±5 µg/m³). Statistical analysis of the data also indicated that there was poor correlation. This was due in part to fluctuating and negative five-minute results generated by Shell.

CONCLUSION

EPA found that the monitoring done by Shell did not have adequate systems and procedures to ensure suitable quality of data being produced.

A review of the data generated by Shell’s benzene monitoring system located at the north-west site between January and June 2005 indicates that the instrument was not operated properly.

A detailed review of the data recorded by Shell’s benzene monitoring system located at the north-west site during March and April 2005 indicates that the high benzene levels recorded were invalid because:

- the statistical parameters generated by the Shell’s benzene monitoring system indicated benzene was not present
- the recorded benzene ultraviolet spectra did not match the benzene reference spectra

EPA REVIEW OF SHELL BENZENE MONITORING

- elevated levels were recorded when the wind was from directions other than the refinery
- EPA monitoring in Corio did not show elevated levels of benzene
- Benzene–toluene ratios were much greater than expected from a petroleum source.

An assessment of the data being generated by EPA's and Shell's systems located side-by-side at the north-east site indicates that data from the Shell system do not correlate with data from the EPA system.

To address these issues Shell has been directed to immediately seek NATA accreditation for the existing benzene monitoring equipment as per licence condition, or to obtain a different monitoring system that can provide reliable benzene monitoring data.

To ensure that reliable concurrent monitoring for benzene is conducted, Shell has been directed by EPA to implement a substitute monitoring program for benzene by using a NATA-accredited method until deficiencies in the operation of their system are addressed. Consistent with this direction Shell has commenced monitoring benzene levels at both the north-west and north-east site using canister systems.

The findings of this review do not change EPA's conclusions that benzene levels in Corio are below national investigation levels and generally similar to those in Melbourne. Benzene levels are expected to drop in 2006 due to the introduction of national cleaner fuel standards.

EPA REVIEW OF SHELL BENZENE MONITORING

APPENDIX

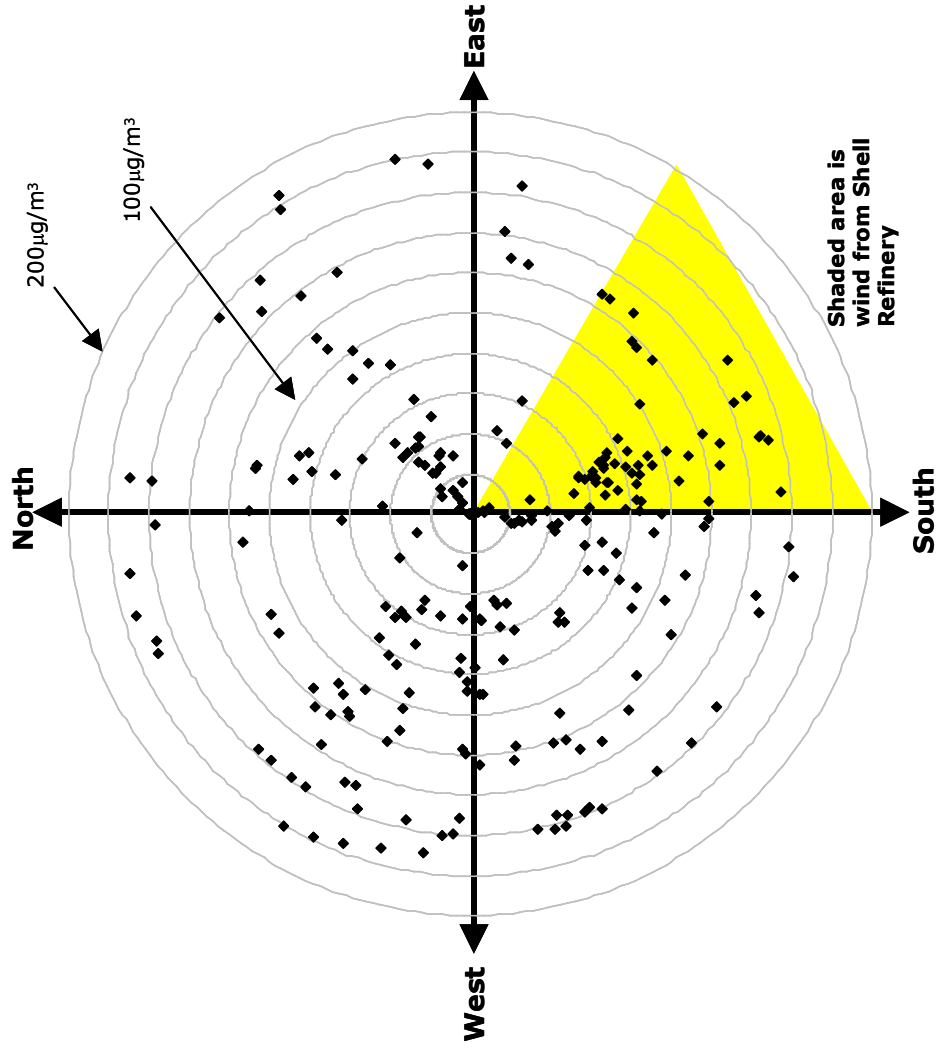


Figure 1: Hourly benzene readings from Shell's benzene monitoring system at the north-west site versus wind direction for results in April 2005

EPA REVIEW OF SHELL BENZENE MONITORING

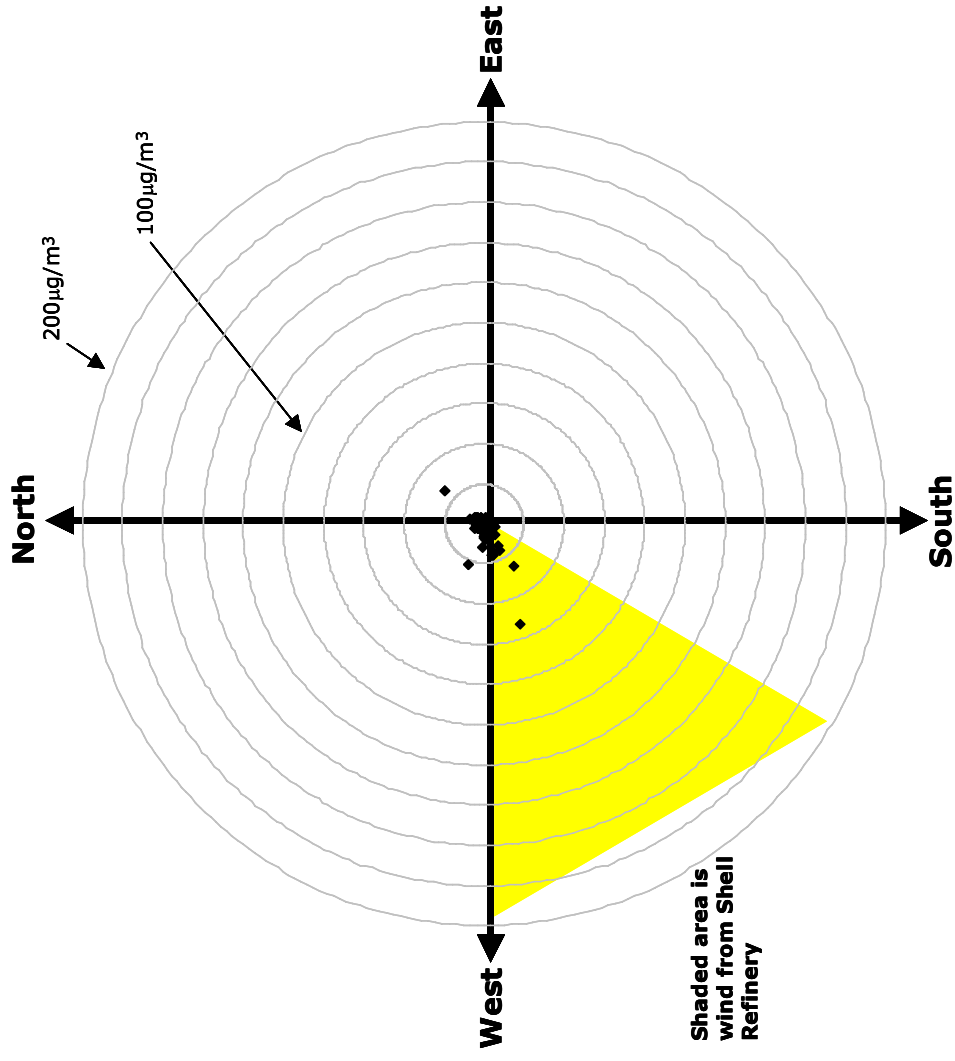


Figure 2: Hourly benzene readings from EPA's monitoring system at the north-east site versus wind direction for results in April 2005

EPA REVIEW OF SHELL BENZENE MONITORING

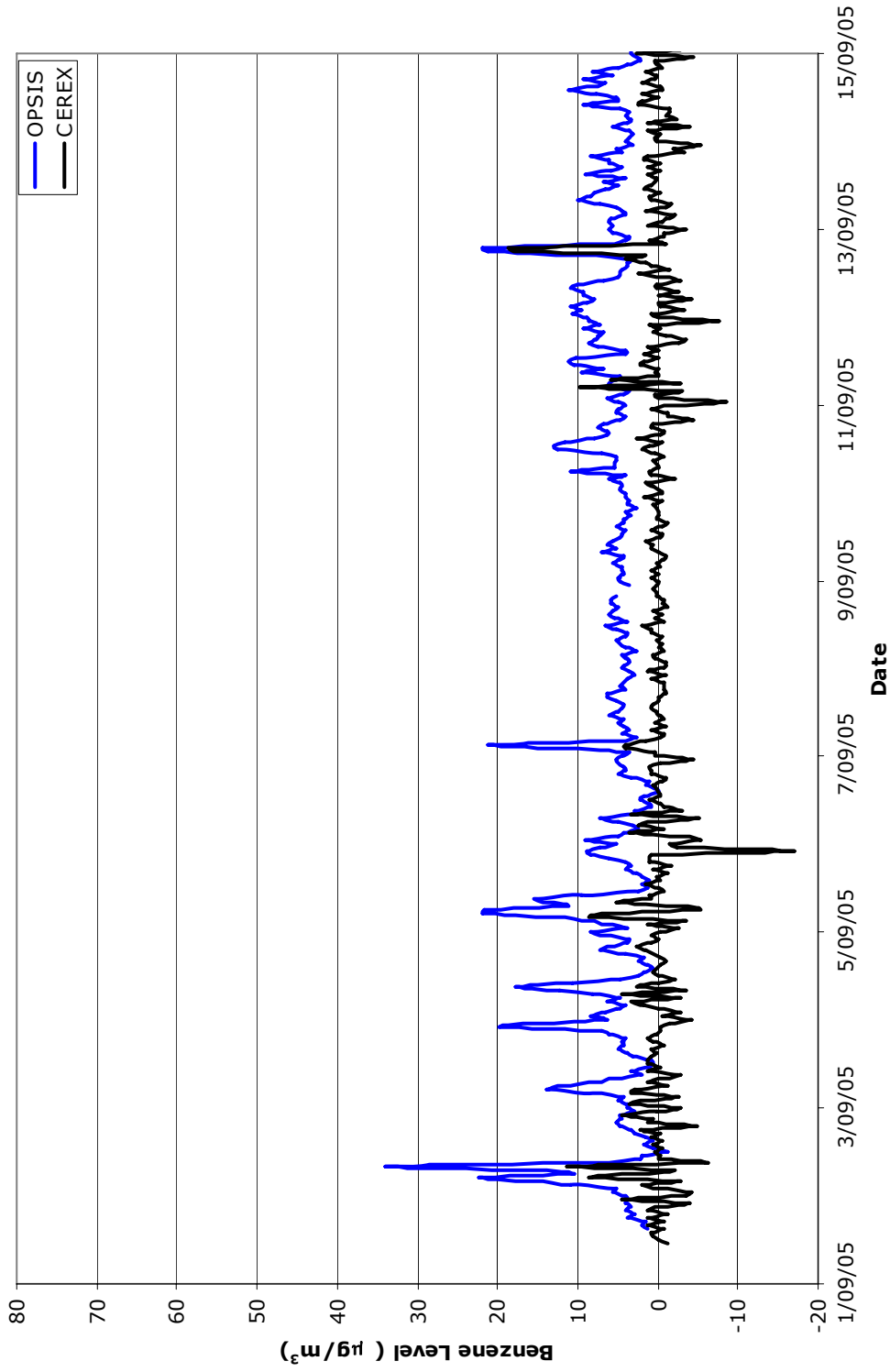


Figure 3: Comparison of Shell's and EPA's hourly benzene levels at the north-east site, September 2005

Community Information

Assessment of the EPA Review of Shell benzene monitoring for March and April 2005

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Friday, September 23, 2005

Summary

The EPA review of the benzene monitoring by Shell for March and April 2005 has been assessed, and found to be valid. There are significant periods within the data record where the calculated benzene amounts are in error due to difficulties with the measurement instrument and software. A detailed reanalysis of the data has not been carried out, but spot checks within the provided data set shows no evidence for extended periods where the benzene concentration was above the “intervention” level.

Background

The primary purpose of this assessment is to consider the approaches used to assess the monitoring data provided by Shell. In particular, this document will examine the arguments for the exclusion of results from a range of periods.

The assessment has been carried out using the information supplied by the EPA, which includes, in addition to the review, the original spectra and reference spectra, the operational and QA logs for the instrument, a draft version of the operating manual and the results of analysis using the software provided by the instrument manufacturer (Cerex).

This assessment is divided into two parts. The first reviews the process used to reject or accept reported benzene concentrations. The second reviews the measurement procedure and highlights difficulties associated with the methodology and reporting

systems used. Note that this second part is not the primary aim of this assessment, and so is not necessarily complete.

Part 1 – Data Assessment

Calculated benzene concentrations have been excluded on the basis of;

1. Low instrumental signal
2. Low statistical confidence in the retrieved amount
3. Poor matching between reference spectra and observed spectra (manual comparison)
4. Assessment of likelihood of benzene emissions based on wind direction, toluene measurements and other monitoring site data.

The low instrumental signal has not been examined by the EPA review, and so will not be commented upon here (It is commented upon in Part 2 of this assessment).

Each of the other exclusion criteria will now be reviewed.

2. Low Statistical confidence in the retrieved amount

The retrieval method used by the software involves the selection of a small spectral window (1-2nm wide) which contains a (strong) absorption feature due to the molecule of interest. I am aware of this method being used with commercial instruments in the infrared. This is not the method used in much of the literature for UV DOAS analysis [Platt, 2000]. (This will be discussed in more depth in the second section of this assessment).

The guidelines provided by Cerex for the interpretation of the fit quality are in line with those used by others in interpreting such fit data. The question does arise as to why the fit quality appears to be, in general, poor. This is discussed in more detail in the second part of this report. Three periods are highlighted in the assessment as rejected for low correlation coefficient. These will be investigated separately.

8-9 March

Retrievals during this period were using as a background the data collected during a calibration. From my attempts at recreating the fitting process it appears that it should have retrieved significant negative concentrations. The use of an inappropriate

background spectrum invalidates the results, and hence the analysis results should be excluded.

20 March 2005

For the period on 20 March where data was rejected, absorbance spectra have been calculated and are shown in Figure 1.

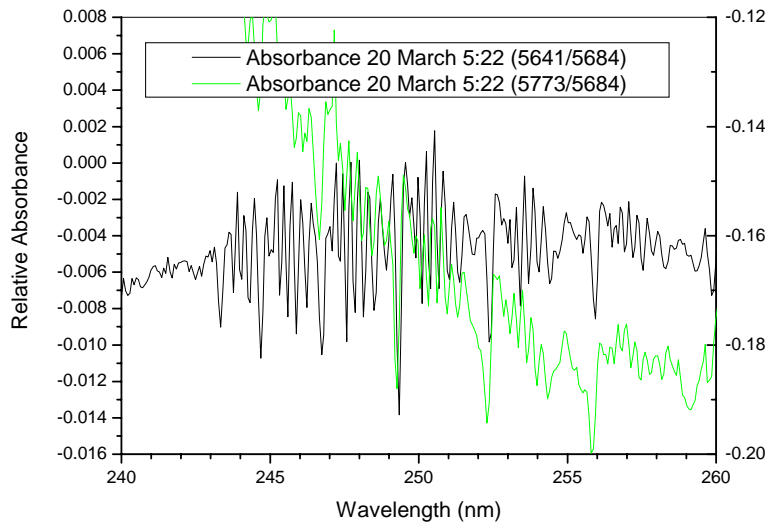


Figure 1 Absorbance spectrum calculated for 20 March 2005.

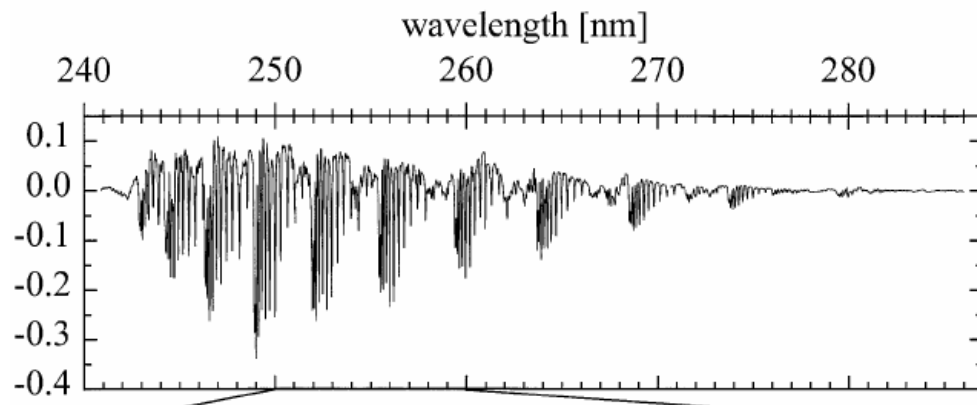


Figure 2 O₂ absorption features[Volkamer, et al., 1998]

Absorbance spectra have been determined using a background taken before and after the period of calculated high benzene. As can be seen, there are a lot of spectral features observable, and they are assignable to absorption due to oxygen (See Figure

2). This will lead to a low correlation coefficient and invalid analysis. These analysis results should be excluded.

3 – 12 April 2005

This data was discounted due to poor background selection and low correlation. An example spectrum is shown below (Figure 3), with a reference spectrum of benzene shown for comparison.

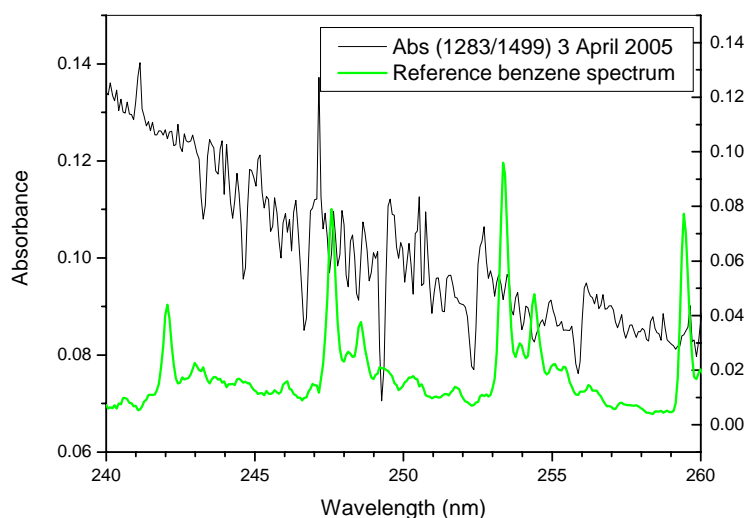


Figure 3 Absorbance spectrum calculated for 3 April 2005 18:47

While there are clear features in the spectrum due to O_2 and possibly some other species, there is no structure that could be clearly assigned to benzene. Further, as the time between the reference and measurement increases there is an apparent increase in spectral “noise”, and hence the likelihood of false benzene estimates increases. During this period a single reference from 3 April was used. Once again the analyses from this period should be disregarded.

3. Poor matching between reference spectra and observed spectra (manual comparison)

Several spectra are shown to highlight the comparison with expected spectra, in a manner similar to that shown above. Note that the spectral region shown in Figure A5 is considerably narrower than those figures shown above. The features observable in figure A5 are due to molecular oxygen (O_2). One important difference is in the

reference benzene spectrum. Shown below (Figure 4) is the comparison spectrum shown in Figure A4 (and supplied to me as a benzene reference spectrum) and the spectra retrieved from the calibration (using the 8 March calibration run). The supplied benzene spectrum is clearly not correct, either in wavelength scale or lineshape. This makes a manual comparison more difficult. Despite this, the conclusion of this section of the review is correct.

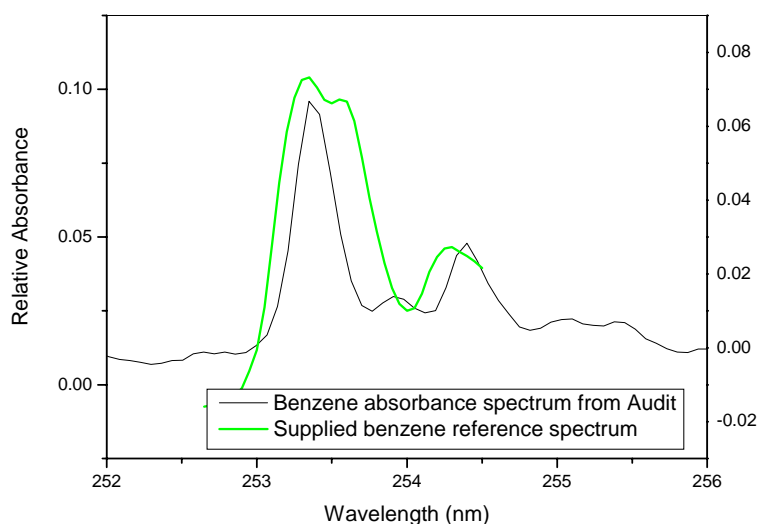


Figure 4 The benzene spectrum recorded during the system audit in March and the supplied reference spectrum for benzene (in green).

4. Assessment of likelihood of benzene emissions based on wind direction, toluene measurements and other monitoring site data.

Firstly, the ratio of benzene to toluene has been considered. There are a number of measurements of this ratio reported in the literature. The molar ratio benzene/ toluene is typically around 0.2 – 0.3 unless aged air is sampled [Chiu, *et al.*, 2005; Fernandes, *et al.*, 2002; Hawas, *et al.*, 2002], when the ratio can approach 1. Once benzene and toluene are released into the atmosphere the ratio can change substantially. Toluene has a much shorter atmospheric lifetime due to its reaction with OH radicals. It should be noted that the retrieval of toluene is in general much more reliable than benzene from the Cerex measurements (median $(r^2_{\text{toluene}})/(r^2_{\text{benzene}}) = 2.6$ for March), in part because the toluene absorption feature used is relatively free from the interference due to O₂.

On this basis, the benzene retrievals for the period 3 April – 14 April are indeed highly suspect. Either the toluene concentrations are substantially underestimated or the benzene concentration is overestimated. Based on the quality of fit argument listed above, and the problems associated with the background listed above, it is highly unlikely that the benzene retrievals are valid.

Secondly, wind direction was also considered. Wind direction, while easy to understand, can be a complex quantity to interpret as air transport depends on the local topography as well as the prevailing wind. It is not clear what wind quantity is used, as there are several ways of averaging wind direction. So I consider that this argument, as presented, supports the conclusions of the assessment, but by itself is not conclusive. However, this is not particularly critical as the measurements themselves are not valid based on the criteria already presented.

The comparison with the other measurement sites is clearly hampered by a lack of data. However, they are broadly consistent with the conclusion that there is no major benzene releases during the periods of high benzene concentrations reported by the Shell monitoring site.

Part 2 – Measurement Methodology

Long path absorption spectroscopy has been used for a number of years to measure a range of compounds in the atmosphere, including benzene and toluene [Eg. Chiu, *et al.*, 2005; Platt, 2000; Eg. Volkamer, *et al.*, 1998]. While the method is sensitive (reported sensitivities to 0.2ppbv for benzene, depending on configuration [Volkamer, *et al.*, 1998]), spectral features due to other atmospheric components, most notably oxygen, complicate the retrieval process. The single beam spectrum, shown in Figure 5, shows large spectral features that are due to atmospheric oxygen, its dimer ($(O_2)_2$) and a collisional complex of oxygen with nitrogen [Volkamer, *et al.*, 1998]. Benzene retrievals must be made in a manner which successfully excludes the impact of these features. For the data shown in Figure 5 the calculated absorbance does successfully eliminate the impact of oxygen absorption. The calculated absorbance spectrum for this period shows clear evidence of benzene.

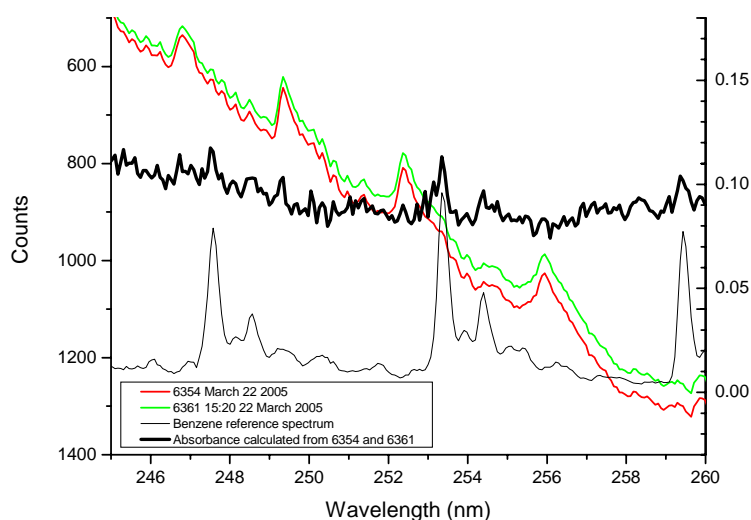


Figure 5 Single beam and calculated absorption spectrum for the Cerex data. Note that there are 4 features in the absorption spectrum that correlate with the benzene spectrum. In contrast to the other spectra shown here the oxygen spectrum is not visible as the viewing conditions are constant between the background and measurement spectrum. The Cerex analysis software only uses the central absorption feature at 253nm.

There are a number of other chemical species that absorb within this region that could be, in principal, determined. This is illustrated by Figure 6 where the absorption spectra for a number of aromatic hydrocarbons and ozone are shown.

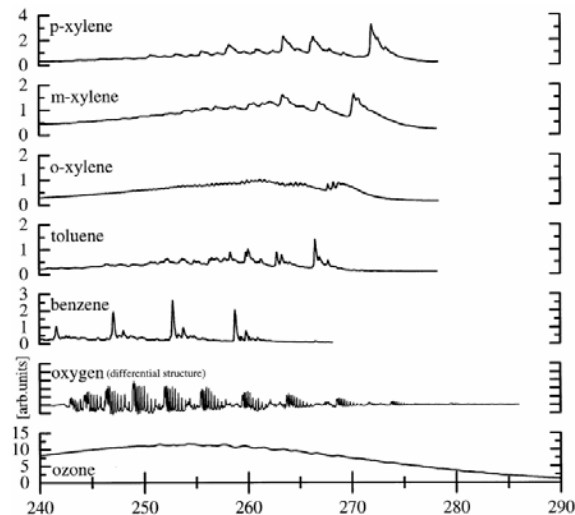


Figure 6 Spectra of various atmospheric components [Volkamer, et al., 1998]

Cerex measurement system

The basic measurement data consists of the average signal recorded over the measurement period for each of the 2048 detector pixels. The magnitude of this signal will depend on a range of factors, including source lamp intensity, atmospheric absorption, optical transmission efficiency in the detection head and sensitivity of the individual detector elements. The manufacturer recommends rejection of all data with low instrument signal. This will be caused primarily by a misalignment of the light source and detection head. An inspection of the data provided showed that the loss of signal tended to be abrupt, consistent with a physical process causing significant misalignment. Inspection of the rejected data in early March showed that the maximum signal reported was around 600, compared with a normal signal level between 2500 and 3500. While it might seem that such a signal may be high enough for some estimate of concentrations, the spectra themselves show a single high value at 278.5nm, and all other pixels have a much lower signal. Following the instrument replacement on March 8/9 this feature disappeared, and the maximum signal was around 60, and appeared to contain no valid spectral features. These “low signal” spectra are clearly not usable for data retrieval and should be excluded.

The remaining measurements are then converted into a useful measure (absorbance) by determining the ratio of the measurement to that of a background spectrum. (There also should be a correction for signal due to the surrounding environment and stray light [Platt, 2000]. Note that this correction appears not to be applied to this data.). Ideally the background spectrum should be determined by measuring the lamp

emission in an identical configuration to the measurement itself, but with the analytes absent. In practice this has been done by choosing periods when it is believed that the air is “clean” (this is determined automatically by the controlling software). As the lamp output varies with time and atmospheric viewing conditions alter, it is important that the background spectrum be measured close in time to the spectrum to be analyzed, and so the background spectrum needs to be regularly updated. This is particularly true for the analysis method employed by Cerex.

As a result of the methodology the calculated amounts are actually the difference between the amounts of the material being determined during the background period relative to the amount present during the measurement. Therefore the determined concentrations may well be negative if a background spectrum has been poorly selected.

Recommendations

1. In the current summary sheets negative concentrations are ignored. They should not be, as it is important that this method reports a difference in concentration.
2. The reference used for analysis is a fundamental piece of information, which needs to be included within the data summaries for each spectral fit.

From the instrument manual it appears that the analysis technique is as follows. For each measurement within the window the reference spectrum (x) is compared with the observational value (y). A linear regression to this data will allow determination of the amount from the slope of the line. The statistical uncertainty in the amount determined and the quality of the fit (r^2) is derived directly from this fit.

There appears to be no attempt made to allow for the oxygen absorption features within the analysis spectral window. This fundamentally limits the accuracy of benzene retrievals. It appears to be the main reason for reduced r^2 retrievals through much of the disputed periods. (For all the spectra shown in figure A5 the structure shown is dominated by oxygen). Note that these oxygen features will change as the amount of oxygen present varies (i.e. with pressure). This will determine the lower limit of benzene concentrations that are retrievable, and this lower limit will depend on atmospheric conditions. For a more reliable retrieval the structure due to oxygen

needs to be explicitly fitted. This is discussed in some detail by Volkamer *et al.* [1998]. Some of the techniques discussed there, such as a high frequency filter to remove the dimer spectral features, may indeed be used by the software, although I can see no evidence of this.

Shown below is an example of this fitting procedure as reported by Volkamer [1998]. The top panel shows the full (filtered) spectrum and the fit for oxygen. The following panels then show the resultant residual and the fit for the components listed.

Recommendation

3. Alternative spectral analysis strategies should be investigated that explicitly calculate the impact of oxygen on the observed spectra.

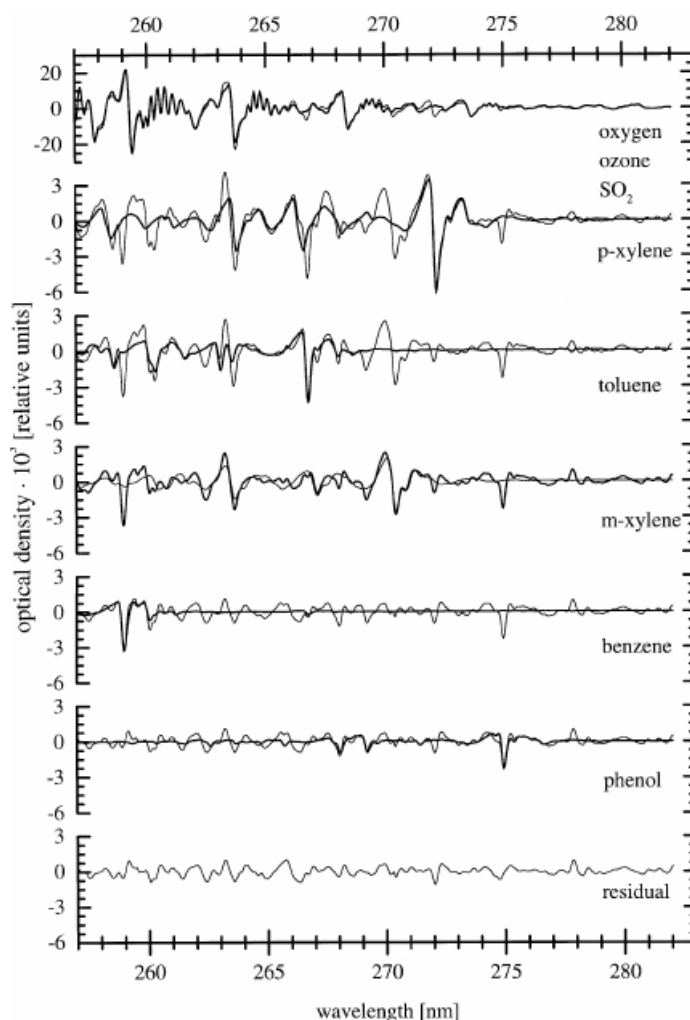


Figure 7 Fitting of a DOAS spectrum following filtering by Volkamer et al (1998).

Instrumental issues

Ten years of personal experience with the spectrometer used within the Cerex instrument has shown them to be very reliable, with consistent spectral sensitivity and good wavelength stability. However, the spectrometer can display a significant temperature dependence, affecting the dark current, spectral sensitivity and wavelength calibration. Currently, the dark signal is monitored as part of the monthly quality control procedure, but it could show a significant diurnal cycle and so more frequent review is required. Ideally the spectrometer should be temperature stabilised (this may be the case already, although there is no evidence of it in the information provided) and the temperature of the spectrometer logged with each measurement.

Recommendations

4. The analysis software should report the dark current estimate as part of the data analysis for each spectrum.
5. The spectrometer temperature should be logged with each spectrum.

References

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