



APPENDIX AVAILABLE ON REQUEST

Research Report 157, Public Health and Air Pollution in Asia (PAPA): Coordinated Studies of Short-Term Exposure to Air Pollution and Daily Mortality in Two Indian Cities

Part 1. Short-Term Effects of Air Pollution on Mortality: Results from a Time-Series Analysis in Chennai, India

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Part 2. Time-Series Study on Air Pollution and Mortality in Delhi

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Appendix D. Determination of Sulphur Dioxide in Ambient Air (Improved West and Gaeke Method)

Note: Appendices Available on the Web appear in a different order than in the original Investigators' Report. HEI has not changed these documents. Appendices were relettered as follows:

Appendix C was originally Appendix I
Appendix D was originally Appendix II
Appendix E was originally Appendix III
Appendix F was originally Annexure 2
Appendix G was originally Annexure 3 (Figure 1)
Appendix H was originally Appendix IV
Appendix I was originally Appendix V
Appendix J was originally Appendix VII

Note: Appendices F & G are for Part 1; Appendices H–J are for Part 2.

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Appendix II: Determination of sulphur Dioxide in Ambient Air

(Improved West and Gaeke Method)

Purpose of experiment: To determine the level of sulphur dioxide (SO₂) in ambient air

Scope: This standard prescribes the methods for the measurement of concentration of sulphur dioxide present in the atmosphere.

INPUTS

Chemicals, required

SI No	Name of the product	Purity of the chemical	Quantity	Formulation
1	0.04M Potassium Tetrachloro mercurate (TCM)	HgCl ₂ : SQ KCl: ExcelsaR EDTA: AR	1L	Dissolve 10.86g mercuric chloride, 0.066g EDTA and 6g potassium chloride and bring to mark in a 1L volumetric flask. It's stable for six months
2	Sulphamic Acid (0.6%)	SQ	100ml	Dissolve 0.6g sulphamic acid in 100ml distilled water. Prepare fresh
3	Formaldehyde (0.2%)	LR	1L	Dilute 5ml formaldehyde solution (36-38%) to 1L with distilled water. Prepare fresh
4	Pararosaniline Stock Solution		100ml	Dissolve 0.5g of pararosaniline in 100ml of distilled water and keep for 2days and filter the stock solution.
5	Pararosaniline Working Solution		250ml	Take 10ml of stock pararosaniline solution and 15ml conc. HCl and dilute to 250ml with distilled water
6	Stock Iodine Solution (0.1N)	SQ	1L	Take 12.7g of iodine in a 250ml beaker, add 40g of potassium iodide and 25ml water. Stir well and dilute to 1L with distilled water.
7	Iodine solution (.01N)	SQ	500ml	Take 50ml of stock iodine solution and dilute to

SI No	Name of the product	Purity of the chemical	Quantity	Formulation
				500ml with distilled water
8	Starch Indicator Solution	LR	200ml	Triturate 0.4g of soluble starch and 0.002g of mercuric iodide preservative with a little water and add the paste slowly to 200ml boiling water. Continue boiling until the solution is clear.
9	Stock sodium thiosulphate solution (0.1N)	LR	1L	Take 25g of sodium thiosulphate in a beaker, add 0.1g of sodium carbonate and dilute to 1L using boiled and cooled distilled water.
10	Sodium thiosulphate Titrant (0.01N)	LR	1L	Dilute 100ml of stock thiosulphate solution to 1L with freshly boiled and cooled distilled water. Allow the solution to stand for one day
11	Standardized Sulphite solution	LR	500ml	Dissolve 0.3g of sodium metabisulphite or 0.4g of sodium sulphite in 500ml boiled and cooled distilled water.
12	Working sulphite-TCM solution	LR	100ml	Measure 2ml of the standard sulphite solution into a 100ml volumetric flask by pipette and bring to mark with 0.04M TCM solution. The solution is stable for 30days if refrigerated at 5°C

Glass ware and other accessories

SI No	Name of the accessory	Quantity	Capacity	Quality
1	Glass impingers	3	35ml	
2	Volumetric flask	5	1L	
3	Volumetric flask	2	500ml	
4	Volumetric flask	1	250ml	
5	Volumetric flask	3	100ml	
6	Volumetric flask	4	50 ml	
7	Volumetric flask	10	25 ml	
8	Beaker		200 ml	
9	Pipette		10ml and 2ml	
10	Burette	1	50ml	

METHODS: Modified West and Gaeke method

Sampling: procedures are described for short-term and long term (30 minutes, 1 hour, 4 hours, 8 hours and 24hours) sampling. One can select different combinations of sampling rate and time to meet special needs. Sample volumes should be adjusted, so that linearity is maintained between absorbance and concentration over the range 0 in question.

30minutes and 1hour sampling: add 10ml of TCM solution to the midget impinger. Collect sample at 1 litre per minute using a rotameter or a critical orifice

4 hours sampling: 30ml TCM solution at 1 liter per minute.

8 hours sampling: 30 ml TCM solution at 0.5 liter per minute

24 hours sampling: 50ml of TCM solution at 0.2 liter per minute

During sampling the impingers should be kept in a ice /cold water tray.

After sampling keep the impingers away from direct sunlight to prevent deterioration.

Measure the flow rate at the time of sampling start and at the end of sampling with the help of rotameter and record the flow rate and start and end time in the data sheet. Determine the volume of air sampled by multiplying the flow rate by the time in minutes. If the analysis is delayed more than a day, sample must be stored in a refrigerator at 5°C.

Storage (if necessary): after sample collection the solutions must be stored at 5°C in a refrigerator. At 22°C losses of sulphur dioxide occur at the rate of 1% per day. If the samples are stored at 5°C for 30 days, no detectable losses of sulphur dioxide occur.

Pre-experiment activities:

Cleaning up of glasswares

Making up of absorbing solution and necessary reagents required for the analysis

EXPERIMENT DETAILS

Sample processing: After collection of sample if a precipitate is observed in the sample, remove it through centrifugation. After sampling, if the final volume of absorbing media is not same as the initial volume, make up to the initial volume by adding distilled water. For 4 hours/8 hour sampling, take 10 ml of exposed absorbing media in clean 25ml volumetric flask. Delay analysis to 20minutes to allow any ozone to decompose. For each set of determinations prepare a reagent blank by adding 10ml of the unexposed TCM solution to a 25ml volumetric flask. To each flask-containing sample or control of reagent blank, add 1 ml of 0.6% sulphamic acid and allow to react for 10 minutes to destroy the nitrite resulting from oxides of nitrogen. Add 2 ml of 0.2% formaldehyde solution and 2ml of para rosaniline (PRA) solution. Bring all flasks to volume of 25ml with distilled water (Millipore) and mix thoroughly. Within 30 to 60 minutes determine the absorbance of the sample, reagent blank and the control solution at 560 nm using cells with a 1 cm path length.

Calculation

$$\text{Concentration of SO}_2 (\mu\text{g}/\text{m}^3) = \frac{(A-A_0) \times B \times V_s \times 10^3}{V_a \times V_t}$$

Where A = Sample Absorbance

A₀ = Reagent blank absorbance

B = Calibration factor, $\mu\text{g}/\text{absorbance}$

V_s = Volume of absorbing reagent used in sampling, ml

10³ = Conversion litres to cubic meters

V_a = Volume of air sampled in litres [average flow rate (lpm) × duration of sampling (min)]

V_t = Aliquot taken for analysis

Preparation of standard graph: Measure by pipette graduated amounts of the working sulphite-TCM solution such as 0, 0.5, 1, 2, 3 and 4 ml into a series of 25 ml volumetric flasks. Add sufficient TCM solution to each flask to bring the volume to approximately to 10 ml. To each flask, add 1 ml of 0.6% sulphamic acid and allow to react for 10 minutes to destroy the nitrite resulting from oxides of nitrogen. Add 2 ml of 0.2% formaldehyde solution and 2ml of pararosaniline solution. Bring all flasks to volume with distilled water (Millipore) and mix thoroughly. Within 30 to 60 minutes determine the absorbance of the sample, reagent blank and the control solution at 560 nm using cells with a 1 cm path length.

Precautions: Wear gloves when handling.

Instrument conditions and operating parameters:

A spectrophotometer suitable for measurement of absorbance at 548 nm with an effective spectral band width of less than 15 nm is required. The wavelength calibration of the instrument should be verified.

OBSERVATION

Expected results:

To get the concentration of sulphur dioxide (SO₂) in microgram per cubic metres in the ambient air

Storage or disposal of the resultant material:

The resulting working sulphite-TCM solution can be stored for 30 days if it is kept in a refrigerator at 5°C.

The resultant solutions, after sufficient dilution with water are disposed through drains.