Regulations 1984 No. 24

Air Pollution Regulations

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SCHEDULE

Regulations 1984 No. 24[1]

Air Pollution Regulations

I, THOMAS UREN, the Minister of State for Territories and Local Government, hereby make the following Regulations under the *Air Pollution Ordinance 1984*.

Dated 19 November 1984.

TOM UREN
Minister of State for Territories and Local Government

PART I—PRELIMINARY

Citation

1. These Regulations may be cited as the Air Pollution Regulations.

Interpretation

2. **(1)** In these Regulations, unless the contrary intention appears—

"equipment" means fuel-burning equipment or industrial plant;

"obscuration" means the ratio of visible light attenuated by pollutants suspended in the effluent stream to incident visible light, expressed as a percentage;

"prescribed date" means the date of commencement of these Regulations;

"process" includes the carrying on of any trade or industry;

"Ringelmann chart" means—

(a) a Ringelmann chart that conforms to British Standards publication BS2742C, as referred to in British Standard BS2742:1969; or

(b) a miniature smoke chart that conforms to British Standards publication BS2742M, as referred to in British Standard BS2742:1969;

"type A socket" means a socket having a clear internal diameter of not less than 108 millimetres and a length of not less than 90 millimetres;

"type B socket" means a socket having a clear internal diameter of not less than 33 millimetres and a length of not more than 50 millimetres.

(2) A reference in these Regulations to a normal volume, in relation to gas, shall be read as a reference to volume corrected to 0° Celsius and to an absolute pressure of 1.013250×10^5 pascal.

(3) A reference in these Regulations to the Bacharach filter paper method shall be read as a reference to a kit having code number 21-7006 and comprising the Bacharach True-Spot Smoke Tester model RCC-B, the Oil Burner Smoke Scale and filter paper, as supplied by the Bacharach Industrial Instrument Company, Pittsburgh, Pennsylvania, U.S.A.

(4) A reference in these Regulations consisting of the words "Australian Standard" followed by letters and figures shall be read as a reference to the standard published under those letters and figures by, or on behalf of, the body incorporated by Royal Charter under the name Standards Association of Australia, being that standard as existing at the date of commencement of these Regulations.

(5) A reference in these Regulations consisting of the words "British Standard" followed by letters and figures shall be read as a reference to the standard published under those letters and figures under the authority of the Executive Board of the body incorporated by Royal Charter under the name British Standards Institution, being that standard as existing at the date of commencement of these Regulations.

(6) A reference in these Regulations consisting of the words "American Standard" followed by letters and figures shall be read as a reference to the standard published under those letters and figures by the American Society for Testing and Materials, being that standard as existing at the date of commencement of these Regulations.

(7) A reference in these Regulations to prescribed obscuration measuring equipment shall be read as a reference to equipment used to measure obscuration, being equipment—

(a) comprising—
  (i) a device which projects light and a receiver which is a photocell detector, mounted on a chimney;
  (ii) transformers, amplifiers, voltage stabilization equipment and calibration adjustment equipment;
  (iii) an indicating meter which is calibrated in percentage obscuration and complies with Australian Standard AS1042-1973;
  (iv) a recorder which produces a permanent record of the obscuration measured and complies with Australian Standard AS1024-1971;
  (v) a visible, external alarm device;
  (vi) an audible, external alarm device; and
  (vii) an alarm actuating device which is either a component part of the obscuration measuring equipment or mounted independently of this equipment; and

(b) having a response time of not greater than 10 seconds, where the response time refers to the time taken, after there has been a change in the level of obscuration in the chimney, for 95% of the corresponding final value of obscuration to be displayed on the indicating meter and recorder.

PART II—STANDARDS FOR EXISTING EQUIPMENT

Application of Part

3. (**1**) This Part applies in relation to air pollution from the carrying on of any process, or the use of any equipment, that is being carried on, or is in use, at the prescribed date.

(**2**) For the purpose of these Regulations, where, on or before the prescribed date, a contract has been entered into for the purchase of equipment, that equipment shall be deemed to be in use at the prescribed date.

Standards of concentration

4. For the purposes of the Ordinance, the following standards of concentration of pollutants are prescribed:

(a) in the case of a process or equipment that emits solid particles, other than a process or equipment referred to in paragraph (b), the total mass of solid particles in each normal cubic metre of residual gases shall not exceed 0.5 gram;

(b) in the case of a process or equipment that consists of a boiler or incinerator that emits solid particles, the total mass of solid particles in each normal cubic metre of residual gases, when adjusted to a basis of 12% carbon dioxide, shall not exceed 0.5 gram;

(c) in the case of equipment that emits smoke, the concentration of smoke shall not exceed—

  (i) in the case of black smoke, a concentration equal to the concentration that appears as Shade 2 on the Ringelmann chart; or

  (ii) in the case of white smoke, 20% obscuration as measured by prescribed obscuration measuring equipment; and

(d) in the case of equipment that uses liquid or gaseous fuel and emits soot, the concentration of soot shall not exceed a concentration equal to the concentration that appears as a blackening index of Shade 3 when tested by the Bacharach filter paper method.

Exceptions

5. (**1**) Notwithstanding regulation 4, the concentration of smoke emitted from equipment—

(a) may exceed the concentration specified in sub-paragraph 4 (c) (i) for a period that does not exceed 20 minutes in any one period of 24 hours where the excessive emission is due solely to the lighting up of a boiler or incinerator from cold if the concentration does not exceed a concentration equal to the concentration that appears as Shade 3 on the Ringelmann chart; and

(b) may exceed the concentration specified in sub-paragraph (1) (a) for a period that does not exceed 10 minutes in any one period of 8 hours where the excessive emission is due solely to the blowing of tubes of a boiler,

if in any case reasonable steps are taken to prevent or minimize the emission.

(2) Notwithstanding paragraph 4 (d), the concentration of soot—

(a) may exceed the concentration specified in paragraph 4 (d) for a period that does not exceed 20 minutes in any one period of 24 hours where the excessive emission is due solely to the lighting up of the equipment from cold if the concentration does not exceed a concentration equal to the concentration that appears as a blackening index of Shade 5 when tested by the Bacharach filter paper method; and

(b) may exceed the concentration specified in paragraph (2) (a) for a period that does not exceed 5 minutes in any one period of 8 hours where the excessive emission is due solely to the blowing of tubes of a boiler,

if in any case reasonable steps are taken to prevent or minimize the emission.

PART III—STANDARDS FOR FUTURE EQUIPMENT

Application of Part

6. (1) This Part applies in relation to air pollution from the carrying on of any process instituted, or the use of any equipment constructed or installed, after the prescribed date.

(2) After the expiration of a period of 6 months after the prescribed date, this Part applies in relation to the carrying on of any process, or the use of any equipment, that was being carried on or that was being used, as the case may be, on the prescribed date, and the provisions of Part II shall, at the expiration of that period, cease to have effect in relation to that process or equipment.

General standards of concentration

7. For the purposes of the Ordinance, the following standards of concentration of pollutants are prescribed:

(a) in the case of a process or equipment that emits solid particles, the total mass of solid particles in each normal cubic metre of residual gases shall not exceed—

   (i) in the case of emissions from a furnace for heating metals, other than a cold blast foundry cupola—0.1 gram; and

   (ii) in the case of emission from any other process or equipment, other than a process or equipment referred to in paragraph (b)—0.25 gram;

(b) in the case of a boiler or incinerator that emits solid particles, the total mass of solid particles in each normal cubic metre of residual gases, when adjusted to a basis of 12% carbon dioxide, shall not exceed—

  (i) in the case of a boiler burning solid fuel—0.25 gram;

  (ii) in the case of an incinerator having a rated capacity of 300 kilograms per hour or less—0.5 gram; and

  (iii) in the case of an incinerator having a rated capacity of more than 300 kilograms per hour—0.25 gram;

(c) in the case of equipment that emits smoke, the concentration of smoke shall not exceed—

  (i) in the case of black smoke, a concentration equal to the concentration that appears as Shade 1 on the Ringelmann chart; or

  (ii) in the case of white smoke, 20% obscuration, as measured by prescribed obscuration measuring equipment; and

(d) in the case of equipment that uses liquid or gaseous fuel and emits soot, the concentration of soot shall not exceed a concentration equal to the concentration that appears as a blackening index of Shade 3 when tested by the Bacharach filter paper method.

Exceptions

8. **(1)** Notwithstanding regulation 7, the concentration of smoke emitted from equipment—

(a) may exceed the concentration specified in sub-paragraph 7 (c) (i) for a period that does not exceed 20 minutes in any one period of 24 hours where the excessive emission is due solely to the lighting up of a boiler or incinerator from cold if the concentration does not exceed a concentration equal to the concentration that appears as Shade 3 on the Ringelmann chart; and

(b) may exceed the concentration specified in paragraph (1) (a) for a period that does not exceed 10 minutes in any one period of 8 hours where the excessive emission is due solely to the blowing of tubes of a boiler,

if in any case reasonable steps are taken to prevent or minimize the emission.

(2) Notwithstanding paragraph 7 (d), the concentration of soot—

(a) may exceed the concentration specified in paragraph 7 (d) for a period that does not exceed 20 minutes in any one period of 24 hours where the excessive emission is due solely to the lighting up of a boiler or incinerator from cold if the concentration does not exceed a concentration equal to the concentration that appears as a blackening index of Shade 5 when tested by the Bacharach filter paper method; and

(b) may exceed the concentration specified in paragraph (2) (a) for a period that does not exceed 5 minutes in any one period of 8 hours where the excessive emission is due solely to the blowing of tubes of a boiler,

if in any case reasonable steps are taken to prevent or minimize the emission.

Specific standards of concentration

9. The amount of pollutant, or of the total of the pollutants, specified in an item in column 2 of the following table, emitted from the source specified in column 3 of that item, shall not exceed the concentration, expressed in amounts per normal cubic metre of residual gases, specified in column 4 of that item opposite that source:

TABLE

Column 1 Item number	Column 2 Substance	Column 3 Source	Column 4 Concentration
1	Sulphuric acid mist and sulphur trioxide	Any process	0.1 g/m³ as sulphur trioxide
2	Nitric acid	Any process manufacturing nitric acid	2.0 g/m³ as nitrogen dioxide
		Any process manufacturing sulphuric acid	1.0 g/m³ as nitrogen dioxide
		Any process other than— (i) a process manufacturing nitric acid or sulphuric acid; or (ii) a gas fired power station	0.5 g/m³ as nitrogen dioxide
3	Oxides of nitrogen	Any process manufacturing nitric acid	2.0 g/m³ as nitrogen dioxide
		Any process manufacturing sulphuric acid	1.0 g/m³ as nitrogen dioxide
		Any process other than— (i) a process manufacturing nitric acid or sulphuric acid; or (ii) a gas fired power station	0.5 g/m³ as nitrogen dioxide
		Gas fired power station	0.35 g/m³ as nitrogen dioxide
4	Carbon monoxide	Any process	1.0 g/m³ as carbon monoxide
5	Fluorine and its compounds	Any process manufacturing aluminium from alumina	0.02 g/m³ as hydrofluoric acid
		Any other process	0.05 g/m³ as hydrofluoric acid
6	Chlorine and its compounds	Any process	0.2 g/m³ as chlorine
7	Hydrogen sulphide	Any process	5 mg/m³ as hydrogen sulphide
8	Antimony and its compounds	Any process	10 mg/m³ as antimony
9	Arsenic and its compounds	Any process	10 mg/m³ as arsenic
10	Cadmium and its compounds	Any process	3 mg/m³ as cadmium
11	Lead and its compounds	Any process	10 mg/m³ as lead
12	Mercury and its compounds	Any process	3 mg/m³ as mercury

Column 1 Item number	Column 2 Substance	Column 3 Source	Column 4 Concentration
13	Antimony, arsenic, cadmium, lead and mercury and their compounds	Any process	10 mg/m^3
14	Acids and acid gases	Any process other than a process manufacturing glazed terra cotta roofing tiles	0.4 g/m^3 as hydrogen chloride

PART IV—TESTING PROCEDURES

Procedure for solid particles

10. **(1)** The determination of the concentration of solid particles in each normal cubic metre of residual gases shall be carried out in accordance with British Standard BS3405:1971 or American Standard ASTM D2928-71.

(2) The number of sampling points in each sampling plane shall be—

(a) in the case of a duct area of less than 0.2 square metre—not less than 4;

(b) in the case of a duct area of not less than 0.2 square metre and not more than 2 square metres—not less than 8; and

(c) in the case of a duct area of more than 2 square metres—1 point for each 0.25 square metre of the cross section of the sampling plane.

(3) The sampling points shall be at the centre of equal areas over the cross-section of the sampling plane.

Procedure for smoke and soot

11. **(1)** The determination of the concentration of smoke in pollutants shall be carried out in accordance with British Standard BS2742:1969.

(2) The determination of the concentration of soot in pollutants shall be carried out in accordance with American Standard ASTM D2156-65.

Measurement and correction of gas volume to normal conditions

12. **(1)** This regulation applies to the measurement, and correction to normal conditions, of gas volumes in the determination of the concentration of pollutants other than solid particles, smoke or soot, being pollutants in respect of which no measurement or correction procedure is specified in the Schedule.

(2) The volume of sample gases taken for the purpose of this regulation shall be measured by passing all the gases drawn through the sampling train into a positive displacement dry meter that complies with the provisions of this regulation.

(3) A meter referred to in sub-regulation (2) shall—

(a) be accurate to within 1%;

(b) have a resolution of 10 millilitres;

(c) have a volume per revolution of 1 litre; and

(d) have a rate of flow of not less than 50 litres per minute.

(4) The volume measured by a meter referred to in sub-regulation (2) shall be corrected to normal conditions.

Manner of testing for other than solid particles

13. A sample of an emission that is to be tested for a substance specified in an item in column 2 of the following table shall be collected and analysed in accordance with the provisions of that part of the Schedule specified in column 3 of the table opposite to that item in column 2:

TABLE

Column 1 Item number	Column 2 Substances	Column 3 Provision in schedule
1	Sulphuric acid mist and sulphur trioxide	Part I
2	Nitric acid	Part II
3	Oxides of nitrogen	Part II
4	Carbon monoxide	Part III
5	Fluorine and its compounds	Part IV
6	Chlorine and its compounds	Part V
7	Hydrogen sulphide	Part VI
8	Antimony and its compounds	Part VII
9	Arsenic and its compounds	Part VII
10	Cadmium and its compounds	Part VII
11	Lead and its compounds	Part VII
12	Mercury and its compounds	Part VII
13	Acids and acid gases	Part VIII

PART V—SAMPLING

Division 1—Sampling of solid particles

Samples of emissions of solid particles

14. (1) Samples of emissions taken for the purposes of determining the amount of solid particles shall be taken through access holes placed and constructed in accordance with Rules 2.2 and 2.3 of British Standard BS3405:1971 and, in addition, shall comply with the provisions of this Division.

(2) Each access hole shall be formed by the use of a type A socket that is fitted with a flange and with a cover, approximately 6 millimetres thick, held in place by 4 bolts, 6 millimetres in diameter, that are equi-spaced on a 150 millimetre pitch circle diameter.

(3) The socket shall be mounted at right angles to the axis of the chimney.

Circular chimneys

15. Each circular chimney shall contain 4 access holes—

(a) that are coplanar and spaced at equal intervals around the chimney;

(b) each centre line of which is directed along a diameter of the chimney; and

(c) that are at the ends of 2 mutually perpendicular sampling lines, one of which is parallel to the plane of the nearest upstream bend.

Rectangular chimneys

16. Each rectangular chimney shall contain 4 access holes—

(a) each 2 of which are on opposite sides of the chimney;

(b) that are coplanar;

(c) that are so located that the centre line of each is a distance of one quarter of the internal length of the side from the centre of that side;

(d) that are located at the ends of 2 parallel sampling lines; and

(e) that are in the plane of the nearest upstream bend.

Division 2—Sampling of other than solid particles

Samples of emissions of other than solid particles

17. (1) Samples of emissions taken for the purposes of determining the content of pollutants other than solid particles shall be taken through access holes placed and constructed in accordance with this Division.

(2) An access hole shall—

(a) in the case of an access hole that exceeds 50 millimetres in length—be formed by the use of a type A socket; or

(b) in the case of an access hole that is 50 millimetres or less in length—be formed by the use of a type B socket,

that is mounted at right angles to the axis of the stack and does not project into the gas stream.

(3) The number of access holes and the type of socket to be used in connection with a sampling line of a length specified in an item in column 1 of the following table is the number and type specified in column 2 of the table opposite to that item in column 1:

Column 1 Length of sampling line	Column 2 Number and type of sockets
Less than 0.75 metre	2 type B
0.75 metre but less than 1.5 metres	4 type B or 2 type A
1.5 metres but less than 2.5 metres	2 type A
2.5 metres or more	4 type A

Division 3—General

Sampling position

18. The sampling position shall be in accordance with Rule 2.2 of British Standard BS3405:1971.

Working platform

19. (**1**) Where—

(a) by virtue of the location of the sampling position, it is not possible, having regard to the safety of the person taking the samples, to take samples through sampling access holes without using a working platform; and

(b) the Authority, by notice in writing served on the occupier of the premises, requires the provision of a working platform,

the occupier of the premises shall provide a working platform that complies with the succeeding provisions of this regulation.

Penalty: $500.

(**2**) The working platform shall—

(a) subject to this sub-regulation, conform to Australian Standard AS1657-1974;

(b) contain a minimum working area of 6 square metres;

(c) have a minimum width—

  (i) in the case of a permanent platform—of 1 metre; and

  (ii) in the case of a scaffolding platform—of 1.25 metres;

(d) be so placed that the inner edge is as close as possible to the chimney;

(e) be so designed that it is not unnecessarily difficult to lift apparatus into position on the platform;

(f) provide access to all access holes on the sampling plane;

(g) be so placed that the access holes are not less than 1.2 metres and not more than 1.75 metres above the floor of the platform; and

(h) be designed to take a load of not less than 300 kilograms.

(**3**) Clearance in the direction of the axis of a socket shall be provided—

(a) in the case of a type A socket—of not less than 3 metres; and

(b) in the case of a type B socket—of not less than 1 metre.

Obligations of occupier

20. (**1**) The occupier of premises shall provide suitable access to the working platform.

(**2**) The occupier of premises shall cause mains current electricity to be available within a distance of 15 metres of a working platform.

Penalty: $500.

PART VI—FUELS

Fuels

21. (**1**) For the purposes of sub-section 23 (2) of the *Air Pollution Ordinance 1984*, sulphur is a prescribed constituent.

(2) The proportion of sulphur present in fuel oil shall be ascertained in accordance with American Standard ASTM D129-64.

(3) The proportion of sulphur that may be present in fuel oil shall not exceed—

(a) in the case of fuel oil used on premises on which there is erected equipment capable of consuming fuel oil, either alone or with another substance, at a rate of not more than 200 kilograms an hour—0.5% by weight; or

(b) in the case of the fuel oil used on premises on which there is erected equipment capable of consuming fuel oil, either alone or with another substance, at a rate of more than 200 kilograms an hour—1.0% by weight.

(4) Notwithstanding sub-regulation (3), fuel oil containing a higher proportion of sulphur than that referred to in paragraph (3) (a) or (3) (b) may be used on premises if the equipment in which the oil is used is fitted with control equipment of such a kind that the emission from the equipment is not greater than the emission that would be emitted from the equipment if fuel oil with a sulphur content of not more than 0.5% or 1.0%, as the case requires, were used.

SCHEDULE

Regulation 13

PART I

TESTING PROCEDURE—SULPHURIC ACID MIST AND SULPHUR TRIOXIDE

1. A gas sample is extracted from a sampling point in the chimney and the acid mist including sulphur trioxide (H_2SO_4 plus SO_3) is separated from sulphur dioxide (SO_2). Both fractions are measured separately by the barium-thorin titration method.

2. (1) The sampling train should consist of the following elements:

(a) Stainless steel nozzle with sharp tapered leading edge;

(b) Pyrex glass probe with a heating system to prevent visible condensation during sampling;

(c) Pyrex glass filter holder;

(d) Four impingers, the first and third of the Greenburg-Smith design with standard tip, the second and fourth of the Greenburg-Smith design modified by replacing the standard tip with a 10 mm internal diameter glass tube extending to 10 mm from the bottom of the impinger flask; during sampling the impingers shall be immersed in an ice bath;

(e) A metering system consisting of a vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 1°C, dry gas meter with 1% accuracy, and related equipment, or equivalent as required to maintain an isokinetic sampling rate and to determine sample volume.

(2) An aneroid type barometer standardized against a Fortin-type barometer shall be used to measure atmospheric pressure to ± 2.5 mm Hg.

(3) A pitot tube shall be attached to the probe to monitor chimney gas velocity.

3. (1) The following items shall be used in sampling:

(a) Filters of glass fibre, or equivalent material, of a suitable size to fit in the filter holder;

(b) Silica gel of an indicating type, 6-16 mesh, dried at 175°C for 2 hours;

(c) Deionized, distilled water;

(d) Isopropanol, 80%, obtained by mixing 800 ml of isopropanol with 200 ml of deionized, distilled water;

(e) Hydrogen peroxide, 3%, obtained by diluting 100 ml of 30% hydrogen peroxide to 1 litre with deionized, distilled water;

(f) Crushed ice.

(2) The following reagents shall be used for sample recovery:

(a) Deionized, distilled water;

(b) Isopropanol, 80%.

(3) The following reagents shall be used for analysis:

(a) Deionized, distilled water;

(b) Isopropanol;

(c) Thorin indicator obtained by dissolving 0.2 g of 1-(o-arsono-phenylazo)-2-napthol-3, 6-disulphonic acid disodium salt (or equivalent) in 100 ml distilled water;

(d) Barium perchlorate (0.01N) obtained by dissolving 1.95 g of barium perchlorate ($Ba(ClO_4)_2.3H_2O$) in 200 ml distilled water and diluting to 1 litre with isopropanol. Standardize with sulphuric acid;

(e) Sulphuric acid standard (0.01N). Standardize to ±0.0002N against 0.01N sodium hydroxide (NaOH) which has previously been standardized against primary standard potassium acid phthalate.

4. (1) The procedure to be followed is as set out in this clause.

(2) Determine the chimney pressure, temperature and range of velocity head.

(3) Preparation of collection train. Place 100 ml of 80% isopropanol in the first impinger, 100 ml of 3% hydrogen peroxide in both the second and third impingers and about 200 g of silica gel in the

SCHEDULE—continued

fourth impinger. Retain a portion of the reagents for use as blank solutions. Assemble the train without the probe in the sequence described in sub-clause 2 (1) with the filter between the first and second impingers. Leak check the sampling train at the sampling site by plugging the inlet to the first impinger and pulling 400 mm Hg vacuum. A leakage rate not in excess of 0.5 litre per minute at a vacuum of 400 mm Hg is acceptable. Attach the probe and turn on the probe heating system. Adjust the probe heater setting during sampling to prevent any visible condensation. Place crushed ice around the impingers. Add more ice during the run to keep the temperature of the gases leaving the last impinger at 20°C or less.

(4) Train operation. For each run record the following data: sampling time, static pressure, chimney temperature, velocity head, pressure differential across orifice meter, gas sample volume, gas temperature, sample box temperature and impinger temperature. Take readings at each sampling point at least every 5 minutes and when significant changes in chimney conditions necessitate additional adjustments in flow rate. To begin sampling, position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Start the pump and immediately adjust the flow to isokinetic conditions. Maintain isokinetic sampling throughout the sampling period. At the conclusion of each run turn off the pump and record the final readings. Remove the probe from the chimney and disconnect it from the train. Drain the ice bath and purge the remaining part of the train by drawing clean ambient air through the system for 15 minutes.

5. (1) The sample shall be recovered in accordance with this clause.

(2) Transfer the isopropanol from the first impinger to a 250 ml graduated cylinder. Rinse the probe, first impinger, and all connecting glassware before the filter with 80% isopropanol. Add the rinse solution to the cylinder. Dilute to 250 ml with 80% isopropanol. Add the filter to the solution, mix and transfer to a suitable storage container. Transfer the solution from the second and third impingers to a 500 ml graduated cylinder. Rinse all glassware between the filter and silica gel impinger with deionized, distilled water and add this rinse water to the cylinder. Dilute to a volume of 500 ml with deionized, distilled water. Transfer the solution to a suitable storage container.

6. (1) The analysis shall be carried out in accordance with this clause.

(2) Shake the container holding isopropanol and the filter. If the filter breaks up, allow the fragments to settle for a few minutes before removing a sample. Pipette a 100 ml aliquot of sample into a 250 ml Erlenmeyer flask and add 2 to 4 drops of thorin indicator. Titrate the sample with barium perchlorate to a pink end point. Repeat the titration with a second aliquot of sample. Shake the container holding the contents of the second and third impingers. Pipette a 25 ml aliquot of sample into a 250 ml Erlenmeyer flask. Add 100 ml of isopropanol and 2 to 4 drops of thorin indicator. Titrate the sample with barium perchlorate to a pink end point. Titrate the blanks in the same manner as the samples.

7. The calibration shall be carried out by standardizing the barium perchlorate with 25 ml of standard sulphuric acid containing 100 ml of isopropanol.

8. To obtain the dry gas volume, correct the sample volume measured by the dry gas meter to normal conditions. Calculate concentrations of sulphuric acid mist (including SO_3) (analysis of first impinger and filter) and sulphur dioxide (analysis of second and third impingers) separately. For acid gases determination add both results.

9. The concentrations of SO_2 and sulphuric acid mist shall be calculated in accordance with the formula—

$$C = \frac{F_n(V_t - V_{tb})\,(N)\left(\frac{V_{soln}}{V_a}\right)}{Vm_{stp}}$$

where—

C is the concentration at normal conditions, dry basis, g/m^3

V_t is the volume of barium perchlorate titrant used for the sample for SO_2 or sulphuric acid mist, ml

V_{tb} is the volume of barium perchlorate titrant used for the blank for SO_2 or sulphuric acid mist, ml

N is the normality of barium perchlorate titrant

V_{soln} is the total solution volume of sulphuric acid mist (first impinger and filter) or sulphur dioxide (second and third impingers), ml

SCHEDULE—continued

V_a is the volume of sample aliquot titrated for SO_2 or sulphuric acid mist, ml

Vm_{stp} is the volume of gas sample through the dry gas meter (normal conditions), m^3

F_n is 4.0×10^{-2} (conversion factor to express both SO_2 and sulphuric acid mist as sulphur trioxide).

PART II

TESTING PROCEDURE—NITRIC ACID AND OXIDES OF NITROGEN

1. A grab sample is collected in an evacuated flask containing a dilute sulphuric acid-hydrogen peroxide absorbing solution and the nitrogen oxides, except nitrous oxide, are measured colorimetrically using the phenoldisulphonic acid (PDS) procedure.

2. (1) The following apparatus should be used:

(a) Pyrex glass heated probe with a filter to remove particulate matter;

(b) T-bore stopcock flask valve connected to a 24/40 standard taper joint;

(c) Pyrex 2 litre collection flask with a round bottom and short neck and 24/40 standard taper opening which is protected against implosion or breakage;

(d) Temperature gauge—dial type thermometer, or equivalent, capable of measuring 1°C intervals from −5°C to +45°C;

(e) Vacuum line made of tubing capable of withstanding a vacuum of 75 mm Hg absolute pressure, with "T" connection and T-bore stopcock, or equivalent;

(f) Pressure gauge comprising a U-tube manometer, 1,000 mm, with 2.5 mm divisions, or equivalent;

(g) One way squeeze bulb;

(h) Pump capable of producing a vacuum of 75 mm Hg absolute pressure.

(2) The apparatus specified in sub-clause (1) shall be assembled in the order in which it is so specified.

(3) An aneroid type barometer standardized against a Fortin type barometer shall be used to measure atmospheric pressure to ± 2.5 mm Hg.

3. The analysis shall be carried out using the following apparatus:

(a) Steam bath;

(b) 250 ml beakers or casseroles, one for each sample and standard (blank);

(c) Volumetric pipettes of 1, 2 and 10 ml capacity;

(d) Transfer pipette—10 ml with 0.1 ml divisions;

(e) Volumetric flasks of 100 ml, one for each sample and the blank and a 1,000 ml flask for the standard;

(f) Spectrophotometer to measure absorbance at 420 nm;

(g) Graduated cylinder of 100 ml with 1.0 ml divisions;

(h) Analytical balance to measure to 0.1 mg.

4. The reagent for sampling should be an absorbing solution. Add 2.8 ml of concentrated H_2SO_4 to 1 litre of distilled water. Mix well and add 6 ml of 3% hydrogen peroxide. Prepare a fresh solution weekly and do not expose to extreme heat or direct sunlight.

5. The sample shall be recovered using the following materials:

(a) Normal sodium hydroxide. Dissolve 40 g of solid sodium hydroxide in 1 litre of water;

(b) Red litmus paper;

(c) Deionized, distilled water.

6. The analysis shall be carried out using the following:

(a) Fuming H_2SO_4 which is from 15% to 18% by weight free sulphur trioxide;

(b) White solid reagent grade phenol;

(c) Concentrated reagent grade sulphuric acid of specific gravity 1.84;

SCHEDULE—continued

(d) Standard solution prepared by dissolving 0.5495 g potassium nitrate (KNO_3) in distilled water and diluting to 1 litre—for the working standard solution, dilute 10 ml of the resulting solution to 100 ml with distilled water. 1 ml of the working standard solution is equivalent to 25 microgram nitrogen dioxide;

(e) Deionized, distilled water;

(f) PDS solution prepared by dissolving 25 g of pure white phenol in 150 ml of concentrated sulphuric acid in a steam bath then cooling and adding 75 ml of fuming sulphuric acid and then heating at 100°C for 2 hours and storing the solution in a dark stoppered bottle.

7. (1) The procedure for sampling shall be carried out in accordance with this clause.

(2) Pipette 25 ml of absorbing solution into a sample flask. Insert the flask valve stopper into the flask with the valve in the "purge" position. Assemble the sampling train and place the probe at the sampling point. Turn the flask valve and the pump valve to their "evacuate" positions. Evacuate the flask to at least 75 mm Hg absolute pressure. Turn the pump valve to its "vent" position and turn off the pump. Check the manometer for any fluctuation in the mercury level. If there is a visible change over the span of one minute check for leaks. Record the initial volume, temperature and barometric pressure. Turn the flask valve to its "purge" position and then do the same with the pump valve. Purge the probe and the vacuum tube using the squeeze bulb. If condensation occurs in the probe and flask valve area heat the probe and purge until the condensation disappears. Then turn the pump valve to its "vent" position. Turn the flask valve to its "sample" position and allow sample to enter the flask for about 15 seconds. After collecting the sample, turn the flask valve to its "purge" position and disconnect the flask from the sampling train. Shake the flask for 5 minutes.

8. (1) The sample shall be recovered in accordance with this clause.

(2) Let the flask set for a minimum of 16 hours and then shake the contents for 2 minutes. Connect the flask to a mercury filled U-tube manometer, open the valve from the flask to the manometer and record the flask pressure and temperature along with the barometric pressure. Rinse the flask with two portions of distilled water (approximately 10 ml) and add rinse water to the sample. For a blank use 25 ml of absorbing solution and the same volume of distilled water as used in rinsing the flask. Add sodium hydroxide drop by drop into both the sample and the blank until alkaline to litmus paper (about 25 to 35 drops in each).

9. (1) The analysis shall be carried out in accordance with this clause.

(2) Transfer the contents to a 250 ml beaker using a small amount of distilled water. Evaporate the solution to dryness on a steam bath and then cool. Add 2 ml of PDS solution to the dried residue, and triturate thoroughly with a glass rod. Ensure that the solution contacts all the residue. Add 1 ml distilled water and four drops of concentrated sulphuric acid. Heat the solution on a steam bath for 3 minutes with occasional stirring. Cool, add 20 ml of distilled water, mix well by stirring and add concentrated ammonium hydroxide drop by drop with constant stirring until alkaline to litmus paper. Transfer the solution to a 100 ml volumetric flask and wash the beaker three times with 4 to 5 ml portions of distilled water. Dilute to the mark and mix thoroughly. If the sample contains solids, transfer a portion of the solution to a clean, dry centrifuge tube and centrifuge or filter a portion of the solution. Measure the absorbance of each sample at 420 nm using the blank solution as a zero. Dilute the sample and the blank with a suitable known amount of distilled water if absorbance falls outside the range of calibration.

10. (1) Calibration shall be carried out in accordance with this clause.

(2) To calibrate the flask volume assemble the flask and flask valve and fill with water to the stopcock. Measure the volume of water to ± 10 ml. Number and record the volume on the flask.

(3) To calibrate the spectrophotometer add 0.0, 4.0, 8.0, 12.0 and 16.0 ml of standard solution to a series of beakers. To each beaker add 25 ml of absorbing solution and add sodium hydroxide drop by drop until alkaline to litmus paper (about 25 to 35 drops). Follow the analysis procedure of clause 9 and use this data to draw a calibration curve of concentration in microgram NO_2 per sample versus absorbance.

SCHEDULE—continued

11. The calculation of sample volume shall be carried out in accordance with the formula—

$$V_{sc} = \frac{T_{std}(V_f - V_a)}{P_{std}}\left(\frac{P_f}{T_f} - \frac{P_i}{T_i}\right)$$

where—

V_{sc} is the sample volume at standard conditions (dry basis), ml

T_{std} is the absolute temperature at normal conditions, 273 Kelvin

P_{std} is the pressure at normal conditions, 760 mm Hg

V_f is the volume of flask and valve, ml

V_a is the volume of absorbing solution, ml

P_f is the final absolute pressure of flask, mm Hg

P_i is the initial absolute pressure of flask, mm Hg

T_i is the initial absolute temperature of flask, Kelvin

T_f is the final absolute temperature of flask, Kelvin

12. (1) The calculation of sample concentration shall be carried out in accordance with this clause.

(2) Read microgram NO_2 for each sample from the plot of microgram NO_2 versus absorbance and divide by corrected sample volume to give concentration in g/m^3.

PART III

TESTING PROCEDURE—CARBON MONOXIDE

1. (1) In this Part—

"accuracy" means the degree of agreement between a measured value and the true value expressed as ± % of full scale;

"DIR" means dispersive infra-red spectrometer;

"GC" means gas chromatograph;

"minimum detectable sensitivity" means the smallest amount of input concentration that can be detected as the concentration approaches zero;

"NDIR" means non-dispersive infra-red analyser;

"precision" means the degree of agreement between repeated measurements of the same concentration expressed as the average deviation of the single results from the mean;

"true value" means the true value obtained by using certified span gases.

(2) The NDIR, DIR and GC carbon monoxide analysers shall, in respect of a feature specified in column 1 of the table in this sub-clause, conform to the specification set out in column 2 of the table opposite to that feature in column 1:

Column 1 Feature	Column 2 Specification
Minimum range	0-1,000ppm
Minimum detectable sensitivity	20ppm
Maximum rise time, 90%, in the case of NDIR and DIR	30 seconds
Maximum fall time, 90%, in the case of NDIR and DIR	30 seconds
Maximum zero drift	10% in 8 hours
Maximum span drift	10% in 8 hours
Minimum precision	2% of full scale
Maximum noise	1% of full scale
Maximum deviation in linearity	2% of full scale
Interference rejection ratio	CO_2—1,000 to 1, H_2O—500 to 1

SCHEDULE—continued

(3) An integrated or continuous gas sample shall be extracted from a sampling point and analysed for carbon monoxide (CO) content using a NDIR, a DIR or an equivalent GC.

2. (1) The method shall be capable of determining carbon monoxide within the range 0 to 1,000 ppm.

(2) The minimum detectable concentration is 20 ppm for a 0 to 1,000 ppm span.

(3) If silica gel and ascarite traps are used to alleviate major interference problems, the measured gas volume must be corrected.

(4) Accuracy shall be ± 5% of span after calibration.

(5) Precision shall be ± 2% of span.

3. (1) The following apparatus shall be used in the case of a continuous sample:

(a) A probe of stainless steel or sheathed Pyrex glass equipped with a filter to remove particulate matter;

(b) An air-cooled condenser or equivalent equipment, to remove any excess moisture;

(c) A dry gas meter.

(2) The following apparatus shall be used in the case of an integrated sample:

(a) A probe of stainless steel or sheathed Pyrex glass equipped with a filter to remove particulate matter;

(b) An air-cooled condenser or equivalent equipment, to remove any excess moisture;

(c) A needle valve, or equivalent, to adjust flow rate;

(d) A pump of the leak-free diaphragm type, or equivalent, to transport gas;

(e) A rotameter, rate meter, or equivalent, to measure a flow range from 0 to 1.0 litre per minute;

(f) A flexible bag, tedlar, or equivalent, with a capacity of 10 to 90 litres per minute. Leak-test the bag in the laboratory before using by evacuating bag with a pump followed by a dry gas meter. When evacuation is complete there should be no flow through the meter;

(g) A pitot tube attached to the probe so that the sampling rate can be regulated proportionally to the chimney gas velocity when velocity is varying with the time or a sample traverse is conducted;

(h) A dry gas meter.

4. (1) The analysis shall be carried out as follows:

(a) For analysis by NDIR or DIR the analysis train shall consist of a filter, a positive displacement volume meter, a drying tube, a carbon dioxide (CO_2) removal tube, a leak-free diaphragm pump, a needle valve, a rate meter and a dry gas meter, connected in that sequence and leading to the analyser. Provision shall be made for injection of zero gas and span gas into the analysis train prior to the drying tube. The drying tube and CO_2 removal tube shall be immersed in an ice water bath;

(b) For analysis by GC the sample zero and span gases shall be transferred to the analyser by a gas-tight syringe or equivalent metering system.

(2) The following materials shall be used:

(a) As a carbon monoxide analyser—a non-dispersive infra-red spectrometer or equivalent;

(b) A drying tube, containing approximately 200 g of silica gel, indicating type, 6 to 16 mesh dried at 175°C for 2 hours;

(c) A filter recommended by NDIR or DIR manufacturer;

(d) A CO_2 removal tube, containing approximately 500 g of ascarite;

(e) An ice water bath for ascarite and silica gel tubes;

(f) A needle valve or equivalent, to adjust flow rate;

(g) A rotameter, rate meter, or equivalent, to measure gas flow rate of 0 to 1.0 litre per minute through NDIR or DIR;

(h) Calibration gases, that is to say, known concentrations of CO in nitrogen for instrument span; the concentrations shall correspond to approximately 60% and 30% span and shall not exceed 1.5 times the applicable emission standard; prepurified grade of nitrogen shall be used for zero;

SCHEDULE—continued

the calibration gases shall be certified by the manufacturer to be within ± 2% of the specified concentration.

5. (1) The procedure to be followed is as set out in this clause.

(2) For continuous sampling, set up the equipment as already described. Place the probe in the chimney at a sampling point and purge the sampling line. Connect the analyser and begin drawing the sample into the analyser. Allow 5 minutes for the system to stabilize, then record the analyser readings as required. CO_2 content of the gas may be determined by weighing the ascarite CO_2 removal and computing CO_2 concentration from the gas volume sampled and the weight gain of the tube.

6. (1) The CO analysis shall be carried out in accordance with this clause.

(2) For analysis using NDIR or DIR assemble the apparatus as already described, calibrate the instrument, and perform other required operations. Purge analyser with N_2 prior to introduction of each sample. Direct the sample stream through the instrument for the test period, recording the readings. Check the zero and span again after the test to assure that any drift or malfunction is detected.

(3) For analysis using GC the analysis system shall be capable of meeting the performance specifications for analysing CO. The system shall be calibrated with the span mixtures before and after the sample determination. The sample shall be injected into the GC and the concentration of CO calculated using accepted GC readout techniques.

7. (1) The calibration shall be carried out in accordance with this clause.

(2) Assemble the apparatus as already described. For analysis using NDIR or DIR allow a minimum time of one hour for warm-up. During this time check the sample conditioning apparatus to ensure that each component is in good operating condition. Zero and calibrate the instrument according to the manufacturer's procedures using, respectively, nitrogen and the calibration gases.

8. (1) The calculation of concentration of carbon monoxide using NDIR or DIR shall be carried out in accordance with this clause.

(2) For analysis using NDIR or DIR calculate the concentration of CO in the stack in accordance with the formula—

$$a = b\,(1 - c)$$

where—

a is the concentration of CO in chimney, ppm by volume (dry basis)

b is the concentation of CO measured by analyser, ppm by volume (dry basis)

c is the volume fraction of CO_2 in sample, that is to say, percent CO_2 from analysis divided by 100.

(3) To convert the concentration of CO in the chimney from ppm by volume to milligrams per normal cubic metre, multiply by 1.25.

PART IV

TESTING PROCEDURE—FLUORINE AND ITS COMPOUNDS

1. The exhaust gases are to be drawn through a polythene sampling probe of approximately 6 mm internal diameter, to two Greenburg-Smith impingers in series, each containing 100 ml of normal sodium hydroxide solution at a rate of not less than 2 litres and not more than 3 litres per minute.

2. The following reagents shall be used:

(a) Citrate buffer solution: 22.63 g of citric acid are to be dissolved in 50 ml of distilled water containing 10 g of sodium hydroxide. The solution is to be diluted with distilled water to a volume of 90 ml and a second solution, containing 20 g of sodium hydroxide dissolved in 100 ml of distilled water, is to be added, drop by drop, to the citrate solution until the pH is between 5.5 and 5.7. The citrate buffer solution is then to be diluted with distilled water to 100 ml.

(b) Preparation of standard fluoride solutions: fluoride standards in the range of 0.1 to 1,000 mg of fluoride ions per litre are to be prepared by diluting with distilled water appropriate quantities of standard sodium fluoride solution to 98 ml. 2 ml of the citrate buffer solution are to be added to each standard sodium fluoride solution. All standard fluoride solutions are to be stored in plastic bottles.

SCHEDULE—continued

3. (1) Analysis shall be carried out in accordance with this clause.

(2) The solutions from the two Greenburg-Smith impingers are to be transferred to a beaker and are to be evaporated to a small volume which is then to be transferred to a platinum crucible. 10 ml of a 4.5% calcium hydroxide solution are to be added and the contents of the crucible are to be evaporated to dryness, then are to be heated for 30 minutes at 150°C before igniting in a muffle furnace at 550°C for 6 hours. 1 gram of powdered silver perchlorate is to be added and the contents are to be transferred to a fluoride distillation apparatus, to which 10 ml of 60% perchloric acid are to be added. The contents are to be steam-distilled at a temperature of 133-137°C, and 240 ml of distillate are to be collected in a volumetric flask of 250 ml capacity and containing 10 ml of the citrate buffer solution. The total distillation time is not to be less than 45 minutes. The analysis of fluoride ion is to be performed by measurement with a specific ion meter in accordance with the manufacturer's instructions and calibrated using the standard fluoride solutions.

PART V

TESTING PROCEDURE—CHLORINE AND ITS COMPOUNDS

Division 1—Determination of chlorine

1. (1) Sampling shall be carried out in accordance with this clause.

(2) The exhaust gases are to be drawn through a Pyrex glass or other non-reactive material probe of 100 mm internal diameter to a midget impinger approximately 110 mm long, 25 mm external diameter and having an orifice of 1 mm internal diameter, containing 10 ml of o-tolidine reagent, by means of a hand exhausting pump. The pump is to have a capacity per stroke of 100 cc (± 1 cc). The pumping strokes are to be made slowly, approximately 10 seconds for each stroke.

(3) To make up o-tolidine reagent, 1 g of o-tolidine is to be dissolved in 100 ml of concentrated hydrochloric acid and the volume is to be made up to 1 litre with distilled water.

(4) To prepare standard colours, 1 g of potassium dichromate is to be dissolved in distilled water containing 1 ml of concentrated sulphuric acid, and the volume is to be made up to 1 litre. A series of standards is to be made by diluting certain quantities of this solution to 10 ml with distilled water as shown in Table 1.

TABLE 1

Standard number	Volume of solution diluted to 10 millilitres
1	8
2	4
3	2
4	1

(5) The standards are to be placed in tubes having the same bore as the midget impinger.

2. (1) The analysis is to be carried out in accordance with this clause.

(2) The colour produced in the midget impinger is to be compared with the standards. The colour of the standard which most closely matches the colour in the midget impinger, together with the number of strokes, gives the chlorine concentration as read from Table 2.

TABLE 2

Standard number	1	2	3	4
Number of strokes	g/m^3	g/m^3	g/m^3	g/m^3
1	0.72	0.36	0.18	0.09
2	0.36	0.18	0.09	0.05
3	0.24	0.12	0.06	0.03

SCHEDULE—continued

Division 2—Determination of Inorganic Chlorine Compounds

1. (1) Sampling is to be carried out in accordance with this clause.

(2) Exhaust gases are to be drawn through two Greenburg-Smith impingers in series, each containing 100 ml of distilled water, at a rate of not less than 2 litres and not more than 3 litres per minute.

2. (1) Analysis is to be carried out in accordance with this clause.

(2) The solutions from the two Greenburg-Smith impingers are to be combined and made up to 250 ml with distilled water. An aliquot of not more than 25 ml is to be taken from the combined solutions from the Greenburg-Smith impingers and is to be added to a 50 ml volumetric flask or Nessler tube, 20 ml of 1 : 1 nitric acid (HNO_3) and 5 ml of decinormal silver nitrate ($AgNO_3$) solution are then to be added. This solution is to be mixed, diluted to the 50 ml mark on the flask, and read immediately in a colorimeter using a filter number 42, that is to say a violet filter having a maximum transmittance near 420 nm, or in a spectrophotometer set to read at 420 nm. The quantity of chloride present is to be read from a standard curve prepared by using the above method with known concentrations of sodium chloride (NaCl). From this result the chloride concentration is to be calculated and expressed as equivalent chlorine.

PART VI

TESTING PROCEDURE—HYDROGEN SULPHIDE

1. Hydrogen sulphide (H_2S) is collected from the source in a series of midget impingers and reacted with alkaline cadmium hydroxide ($Cd(OH)_2$) to form cadmium sulphide (CdS). The precipitated CdS is then dissolved in hydrochloric acid and absorbed in a known volume of iodine solution. The iodine consumed is a measure of the H_2S content of the gas. An impinger containing hydrogen peroxide is included to remove sulphur dioxide (SO_2) as an interfering species.

2. (1) The sampling train shall consist of the following elements:

(a) A sampling line of 6-7 mm Teflon tubing to connect sampling train to sampling valve, with provisions for heating to prevent condensation. A pressure reducing valve prior to the Teflon sampling line may be required depending on sampling stream pressure;

(b) Five midget impingers each with 30 ml capacity, or equivalent, immersed in an ice bath container to maintain absorbing solution at a constant temperature;

(c) Silica gel drying tube, to protect pump and dry gas meter;

(d) A needle valve, or equivalent, of stainless steel or other corrosion resistant material, to adjust gas flow rate;

(e) A leak free pump, of the diaphragm type, or equivalent, to transport gas. (This is not required if the sampling stream is under positive pressure.);

(f) A rotameter, rate meter, or equivalent, to measure a flow rate of 0 to 3 litres per minute;

(g) A dry gas meter, sufficiently accurate to measure sample volume to within 1%.

(2) An aneroid type barometer standardized against a Fortin-type barometer shall be used to measure atmospheric pressure to ± 2.5 mm Hg.

3. (1) The following reagents shall be used in sampling:

(a) An absorbing solution of $Cd(OH)_2$. Mix 4.3 g cadmium sulphate hydrate ($3 CdSO_4.8H_2O$) and 0.3 g of sodium hydroxide (NaOH) in 1 litre of distilled water. Mix well. $Cd(OH)_2$ formed in this mixture will precipitate as a white suspension. Therefore this solution must be thoroughly mixed before using to ensure an even distribution of the $Cd(OH)_2$.

(b) Hydrogen peroxide, 3 per cent. Dilute 30% hydrogen peroxide to 3% as needed. Prepare fresh daily.

(c) Silica gel of an indicating type, 6-16 mesh, dried at 175°C for 2 hours.

(2) The following reagents shall be used in sample recovery:

(a) Hydrochloric acid solution (HCl 10% by weight). Mix 230 ml of concentrated HCl (specific gravity 1.19) and 770 ml of distilled water.

(b) Iodine solution, 0.1 N. Dissolve 24 g potassium iodide (KI) in 30 ml of distilled water in a 1 litre graduated cylinder. Weigh 12.7 g of resublimed iodine (I_2) into a weighing bottle and add

SCHEDULE—continued

to the KI solution. Slowly dilute the solution to 1 litre with distilled water with swirling. Filter the solution, if cloudy, and store in a brown glass-stoppered bottle.

(c) Standard iodine solution, 0.01N. Dilute 100 ml of the 0.1N iodine solution in a volumetric flask to 1 litre with distilled water. Standardize daily as follows: Pipette 25 ml of the 0.01N iodine solution into a 125 ml conical flask. Titrate with sodium thiosulphate solution, standard 0.01N until the solution is a light yellow. Add a few drops of starch indicator solution and continue titrating until the blue colour just disappears. From the results of this titration calculate the exact normality of the iodine solution.

(d) Distilled, deionized water.

(3) The following reagents shall be used in analysis:

(a) Sodium thiosulphate solution, standard 0.1N. For each litre of solution, dissolve 24.8 g of sodium thiosulphate ($Na_2S_2O_3.5H_2O$) in distilled water and add 0.01 g of anhydrous sodium carbonate (Na_2CO_3) and 0.4 ml of chloroform ($CHCl_3$) to stabilize. Mix thoroughly by shaking or by aerating with nitrogen for approximately 15 minutes and store in a glass-stoppered glass bottle. Standardize frequently as follows: Weigh into a 500 ml volumetric flask about 2 g of potassium dichromate ($K_2Cr_2O_7$) weighed to the nearest milligram and dilute to the 500 ml mark with distilled water. Use dichromate which has been crystallized from distilled water and oven-fried at 182°C to 199°C. Dissolve approximately 3 g of KI in 50 ml of distilled water in a glass-stoppered 500 ml conical flask, then add 5 ml of 20% HCl solution. Pipette 50 ml of the dichromate solution into this mixture. Gently swirl the solution once and allow it to stand in the dark for 5 minutes. Dilute the solution with 100 to 200 ml of distilled water washing down the sides of the flask with part of the water. Swirl the solution slowly and titrate with the thiosulphate solution until the solution is light yellow. Add 4 ml of starch solution and continue with a slow titration with the thiosulphate until the bright blue colour has disappeared and only the pale green colour of the chromic ion remains. From this titration, calculate the exact normality of the $Na_2S_2O_3$ solution.

(b) Sodium thiosulphate solution, standard 0.01N. Pipette 100 ml of the standard 0.1N thiosulphate solution into a volumetric flask and dilute to one litre with distilled water.

4. (1) The procedure for sampling shall be in accordance with this clause.

(2) Assemble the sampling train in the order specified in sub-clause (1) with the five midget impingers in series. Place 15 ml of 3% hydrogen peroxide in the first impinger. Place 15 ml of the absorbing solution in each of the next three impingers leaving the fifth dry. Place crushed ice around the impingers. Add more ice during the run to keep the temperature of the gases leaving the last impinger at about 20°C or less.

(3) Purge the connecting line between the sampling valve and the first impinger. Connect the sample line to the train. Record the initial reading on the dry gas meter.

(4) Open the flow control valve and adjust the sampling rate to 1.1 litres per minute. Read and record the meter temperature.

(5) Continue sampling for a minimum of 10 minutes. At the end of the sample time close the flow control valve and read the final meter volume and temperature.

(6) Disconnect the impinger train from the sampling line. Purge the train with clean ambient air for 15 minutes to ensure that all H_2S is removed from the hydrogen peroxide. Cap the open ends and move to the sample clean-up area.

5. (1) The procedure for sample recovery shall be in accordance with this clause.

(2) Pipette 50 ml of 0.01N iodine solution into a 250 ml beaker. Add 50 ml of 10% HC1 to the solution. Mix well.

(3) Discard the contents of the hydrogen peroxide impinger. Carefully transfer the contents of the remaining four impingers to a 500 ml iodine flask.

(4) Rinse the four absorbing impingers and connecting glassware with three portions of the acidified iodine solution. Use the entire 100 ml of acidified iodine for this purpose. Immediately after pouring the acidified iodine into an impinger, stopper it and shake for a few moments before transferring the rinse to the iodine flask. Do not transfer any rinse portion from one impinger to another; transfer it directly to the iodine flask. Once acidified iodine solution has been poured into any glassware containing CdS sample, the container must be tightly stoppered at all times except when adding more solution and this must be done as quickly and carefully as possible. After adding any

SCHEDULE—continued

acidified iodine solution to the iodine flask, allow a few minutes for absorption of the H_2S into the iodine before adding any further rinses. Titrate the blanks in the same manner as the samples.

(5) Follow this rinse with two more rinses using distilled water. Add the distilled water rinses to the iodine flask. Stopper the flask and shake well. Allow about 30 minutes for absorption of the H_2S into the iodine, then complete the analysis titration. The iodine flask should be kept stoppered except when adding sample or titrant.

(6) Prepare a blank in an iodine flask using 45 ml of the absorbing solution, 50 ml of 0.01 iodine solution and 50 ml of 10% HCl. Stopper the flask, shake well and analyse with the samples.

6. (1) The analysis shall be carried out in accordance with this clause.

(2) This analysis titration should be conducted at the sampling location in order to prevent loss of iodine from the sample. Titration should never be made in direct sunlight.

(3) Titrate the solution in the flask with 0.01 N $Na_2S_2O_3$ solution until the solution is light yellow. Add 4 ml of the starch indicator solution and continue titrating until the blue colour just disappears.

7. The calculation of the concentration of H_2S in the gas stream at normal conditions shall be made by using the formula—

$$a = b\,\frac{(C_I d_I - C_T d_T)\text{ sample} - (C_I d_I - C_T d_T)\text{ blank}}{e}$$

Where—

a is the concentration of H_2S at normal conditions, g/m^3

b is the conversion factor, that is to say 170

C_I is the volume of standard iodine solution, ml

d_I is the normality of standard iodine solution

C_T is the volume of standard sodium thiosulphate solution, ml

d_T is the normality of standard sodium thiosulphate solution

e is the normal dry gas volume, m^3.

PART VII

TESTING PROCEDURES—ANTIMONY, ARSENIC, CADMIUM, LEAD, MERCURY AND THEIR COMPOUNDS

1. (1) When sampling for lead, antimony, cadmium and arsenic and their compounds the procedures outlined below are to be followed except where the particles present are greater than 1 micron in diameter. In that event, the isokinetic methods outlined for testing solid particles should be followed, provision being made for the collection of any fumes not collected by the apparatus. Samples so collected shall be digested and analysed in the media and using the methods outlined below.

2. (1) The analysis of lead, antimony and cadmium shall be carried out in accordance with this clause and clauses 3 to 10 (inclusive).

(2) A sample shall be absorbed in a normal nitric acid solution and analysed for each of the metals by conventional atomic absorption techniques. When organo-metallic compounds are present an absorbing solution of greater oxidizing power is necessary.

(3) The sampling train shall consist of the following elements:

(a) A nozzle of any suitable material substantially resistant to chemical attack by components of the gas stream. The leading edge of the nozzle shall be sharp and tapered;

(b) A probe of any suitable material substantially resistant to chemical attack by components of the gas stream. The probe shall have the necessary mechanical strength and heat resistance and shall be fitted with a heating system to prevent condensation during sampling. The internal dimensions of the probe shall permit a flow rate through the impingers within the range from 20 to 30 litres per minute for isokinetic sampling;

(c) Impingers, the first and second of which shall be of the Greenburg-Smith design and the third of which shall be of the Greenburg-Smith design modified by replacing the standard tip with a 10 mm internal diameter tube extending to 10 mm from the bottom of the impinger flask;

SCHEDULE—continued

(d) A metering system comprising a vacuum gauge, leak-free pump, thermometers capable of measuring to within 1°C, dry gas meter of 1% accuracy and related equipment, or their equivalents, as required to maintain the isokinetic sampling rate and determine sample volume.

(4) The apparatus specified in sub-clause (3) shall be assembled in the order in which it is so specified.

(5) An aneroid type barometer standardized against a Fortin type barometer shall be used to measure atmospheric pressure to ± 2.5 mm Hg.

(6) A pitot tube of type S or its equivalent shall be attached to the probe to monitor chimney gas velocity.

3. (1) The following items shall be used in sampling:

(a) Silica gel of an indicating type, 6-16 mesh, dried at 175°C for 2 hours;

(b) An absorbing solution of normal nitric acid. For a solution of greater oxidizing power 3 g of potassium permanganate shall be added to each litre of nitric acid used. This mixture shall be used immediately and not stored;

(c) Crushed ice.

4. Glass distilled water shall be used in sample recovery.

5. The following reagents shall be used in analysis:

(a) Glass distilled water;

(b) Hydrogen peroxide, 30%—this solution is only required for absorbing solutions containing potassium permanganate;

(c) Hydroxylamine hydrochloride solution. Dissolve 50 g of hydroxylamine hydrochloride solution in 100 ml of glass distilled water—this solution is only required for absorbing solutions containing potassium permanganate;

(d) Sulphuric acid—concentrated pure grade of specific gravity 1.84;

(e) The following primary standard solutions containing 1g/litre of lead, antimony or cadmium shall be prepared by dissolving the stated reagent and making the resultant solution to 1 litre with glass distilled water:

(i) standard lead solution—dissolve 1.599 g of analytical reagent grade lead nitrate in 500 ml of normal nitric acid and dilute to 1 litre with glass distilled water;

(ii) standard antimony solution—dissolve 2.699 g of analytical reagent grade antimony potassium tartrate in glass distilled water and dilute to 1 litre with glass distilled water;

(iii) standard cadmium solution—dissolve 2.282 g of analytical reagent grade cadmium sulphate in 500 ml of normal hydrochloric acid and dilute to 1 litre with glass distilled water.

6. A series of working standards shall be prepared using appropriate dilutions of the primary standard solutions and the atomic absorption spectrometer shall be calibrated against those solutions. The wavelength selected for each metal for this calibration of the spectrometer will determine the range of working standards required. The wavelength shall be selected in accordance with the instructions given by the manufacturer of the atomic absorption spectrometer to provide the required sensitivity for measuring the concentration of metal ions collected in the absorbing solution.

7. (1) The procedure for sampling shall be in accordance with this clause.

(2) Determine the minimum number of sampling points for isokinetic sampling and measure the chimney pressure, temperature, moisture and range of velocity head. Place 75 ml of the absorbing solution in each of the first and second impingers, and about 200 g of silica gel in the third impinger. Retain a 150 ml portion of the absorbing solution for use as a blank solution. Leak check the sampling train at the sampling site by plugging up the inlet to the first impinger and pulling a 400 mm Hg vacuum. A leakage rate not in excess of 0.5 litre per minute is acceptable. Attach the probe and probe heater and adjust the probe heater to prevent condensation during sampling. Place crushed ice around the impingers. Add more ice during the run to keep the temperature of the gases leaving the last impinger at 20°C or less.

8. (1) The operation of the sample train shall be carried out in accordance with this clause.

SCHEDULE—continued

(2) Take readings at each sampling point at least every 5 minutes and when significant changes in chimney conditions necessitate additional adjustments in flow rate. Maintain isokinetic sampling throughout the sampling period. Drain the icebath and purge the remaining part of the train by drawing clean ambient air through the system for 15 minutes.

9. (1) The procedure for sample recovery shall be in accordance with this clause.

(2) Remove the absorbing solution from the first and second impingers and place each in a glass sample storage container. Rinse the impingers and all connecting glassware with glass distilled water and add the rinsings to the storage container.

10. (1) The analysis shall be carried out in accordance with this clause.

(2) Metal ion sample solution. Transfer the absorbing solution to a 1 litre volumetric flask and dilute to volume with glass distilled water. If potassium permanganate has been added to the absorbing solution, transfer the contents of the impingers to a 250 ml beaker, add 25 ml of the sulphuric acid and evaporate carefully to the first appearance of white sulphuric acid fumes. Dilute the solution to approximately 150 ml and add hydrogen peroxide solution carefully and with constant stirring until the permanganate colour just disappears. Add 1 ml of hydroxylamine hydrochloride solution and heat just to boiling and then cool. Should a white precipitate of lead sulphate appear, dilute, neutralize with ammonium hydroxide, then add sufficient 50% lead free ammonium citrate solution to dissolve precipitate. Dilute the solution to 1 litre.

(3) Follow the same procedure for the blank solution.

(4) Analysis for metal. Remove an aliquot of the metal ion solution and measure the absorption of each metal at the appropriate wavelength using an atomic absorption spectrometer. Repeat the operation for the blank solution and determine the quality of metal ion in the solution from the appropriate calibration curve. If the sample solution gives an absorption greater than the upper limit of the range of the calibration curve, make appropriate dilutions to the solution and remeasure the absorption.

11. (1) The determination of arsenic shall be in accordance with this clause.

(2) The exhaust gases are to be drawn through two Greenburg-Smith impingers in series, each containing 100 ml of decinormal sodium hydroxide at a rate not less than 2 litres and not more than 3 litres per minute.

(3) The reagents to be used are:

(a) Lead acetate solution: 10 g of Pb $(C_2H_3O_2)_2.3H_2O$ are to be dissolved in 100 ml of distilled water; and

(b) Silver diethyldithiocarbamate: 1 g of $AgSCSN(C_2H_5)_2$ is to be dissolved in 200 ml of pyridine.

(4) The collected arsenic is to be reduced to arsine and is to be passed through glass wool impregnated with lead acetate solution to an absorber tube. Arsenic forms a soluble red complex and the intensity of the soluble red complex so formed is to be measured photometrically at 535 nm. The colour intensity, as measured, is to be compared with a standard curve prepared from the colours produced by 0 to 10 micrograms of arsenic.

12. (1) The determination of mercury shall be in accordance with this clause.

(2) The determination of mercury vapour or fume less than 1 micron in diameter is to be carried out by sampling the exhaust gases through a mercury vapour photoelectric detector calibrated according to the manufacturer's instructions. The absorbance is to be read at 253.7 nm.

(3) The determination of mercury greater than 1 micron in diameter shall be in accordance with sub-clauses (4), (5) and (6).

(4) The exhaust gases are to be drawn through two Greenburg-Smith impingers in series, each containing 100 ml of an aqueous solution containing 25 ml of concentrated nitric acid, 2 ml of concentrated sulphuric acid and 1 g of potassium permanganate.

(5) The excess permanganate and manganese dioxide is to be removed by the drop by drop addition of 30% hydrogen peroxide. The dissolved oxygen is to be expelled by boiling. 0.5 g of hydroxylamine hydrochloride is to be added, and the mercury complex is to be extracted from the solution by shaking with successive portions of a chloroform solution of diphenylthiocarbazone containing 25 mg per litre, until the diphenylthiocarbazone solution is present in excess. The chloroform extract is to be treated with 50 ml of water and 2 ml of 5% potassium permanganate solution, and 2 ml of normal sulphuric acid. Sufficient 10% sodium nitrate is to be added to the aqueous

SCHEDULE—continued

solution to react with the excess permanganate, the free nitrous acid left by the addition of the hydroxylamine hydrochloride is to be destroyed by heating just to boiling.

(6) The solution is to be titrated in a separatory funnel with a carbon tetrachloride solution of diphenylthiocarbazone containing 1.25 mg of diphenylthiocarbazone per litre. Small portions of this solution are to be added until it is in excess. A standard mercuric nitrate solution, containing 10 mg of mercury per litre, is to be used for back titration of the excess. The strength of the diphenylthiocarbazone solution is to be determined each time it is used, by treating a solution containing 0.1 mg of mercury, as mercuric nitrate, in the same manner as described for the test sample. To ascertain the quantity actually present in the material analysed, the mercury found in the blank is to be subtracted from that found in the test sample.

PART VIII

TESTING PROCEDURE—ACIDS AND ACID GASES

1. The gas sample is to be withdrawn from the sampling point in the chimney in such a way that the velocity of the gases entering the sampling nozzle placed in the chimney is, as nearly as practicable, the same velocity as the gases flowing in the chimney at the sampling point.

2. (1) The sampling train should consist of the following elements:

(a) Stainless steel nozzle with sharp tapered leading edge;

(b) Pyrex glass probe with a heating system to prevent visible condensation during sampling;

(c) Pyrex glass filter holder;

(d) Two impingers of the Greenburg-Smith design; during sampling the impingers shall be immersed in an ice bath;

(e) A metering system consisting of a vacuum gauge, leak-free pump, thermometers capable of measuring temperature to within 1°C, dry gas meter with 1% accuracy, and related equipment, or equivalent as required to maintain an isokinetic sampling rate and to determine sample volume.

(2) An aneroid type barometer standardized against a Fortin-type barometer shall be used to measure the atmospheric pressure to ± 2.5 mm Hg.

(3) A pitot tube shall be attached to the probe to monitor chimney gas velocity.

3. (1) The following items shall be used in sampling:

(a) Filters of glass fibre, or equivalent material, of a suitable size to fit in the filter holder;

(b) Silica gel of an indicating type, 6-16 mesh, dried at 175°C for 2 hours;

(c) Deionized, distilled water;

(d) Sodium tetraborate solution; 19.071 g of sodium tetraborate decahydrate are to be dissolved in deionized, distilled water and the volume adjusted to 1 litre with deionized, distilled water.

4. (1) The procedure to be followed is set out in this clause.

(2) Determine the chimney pressure, temperature and range of velocity head.

(3) Preparation of collection train: Place 100 ml of deionized, distilled water in each impinger. Assemble the train without the probe in the sequence described in sub-clause 2 (1) with the filter between the first and second impingers. Leak check the sampling train at the sampling site by plugging the inlet in the first impinger and pulling 400 mm Hg vacuum. A leakage rate not in excess of 0.5 litre per minute at a vacuum of 400 mm Hg is acceptable. Attach the probe and turn on the probe heating system. Adjust the probe heater setting during sampling to prevent any visible condensation. Place crushed ice around the impingers. Add more ice during the run to keep the temperature of the gases leaving the last impinger at 20°C or less.

(4) Train operation. For each run record the following data: sampling time, static pressure, chimney temperature, velocity head, pressure differential across orifice meter, gas sample volume, gas temperature, sample box temperature and impinger temperature. Take readings at each sampling point at least every 5 minutes and when significant changes in chimney conditions necessitate additional adjustments in flow rate. To begin sampling, position the nozzle at the first traverse point with the tip pointing directly into the gas stream. Start the pump and immediately adjust the flow to isokinetic conditions. Maintain isokinetic sampling throughout the sampling period. At the conclusion of each run turn off the pump and record the final readings. Remove the probe from the chimney and

SCHEDULE—continued

disconnect it from the train. Drain the ice bath and purge the remaining part of the train by drawing clean ambient air through the system for 15 minutes.

(5) Analysis: The contents of the impingers are to be filtered and transferred to a beaker and the contents brought to boiling point. The volume is to be adjusted to 250 ml with demineralized water. An aliquot consisting of 100 ml is to be titrated with sodium tetraborate solution using methyl red-methylene blue as the indicator.

NOTE

1. Notified in the *Commonwealth of Australia Gazette* on 28 November 1984.