

MDHS

*Methods for the Determination of
Hazardous Substances*
Health and Safety Laboratory



85

Triglycidyl isocyanurate (and coating powders containing triglycidyl isocyanurate) in air

Laboratory method using pumped filter,
desorption and liquid chromatography

June 1997

INTRODUCTION

Scope

1 This method covers the analysis of time-weighted average concentrations of triglycidyl isocyanurate (TGIC), premix and coating powders containing TGIC. Premix is defined as formulated and mixed coating powder prior to extrusion in manufacture and coating powder as the extruded and milled powder supplied by manufacturing companies to users. Both 'free' or unbound TGIC and cross-linked TGIC will be present in the coating powders as some TGIC cross-links during the manufacturing process - this method measures only 'free' TGIC in coating powders.^{1,2} The extent of cross-linking varies for different powders, and if it is desirable to know the proportion of 'free' in a series of coating powders each coating powder must be analysed separately.

2 HSE booklet MDHS 14³ gives advice on sampling for total inhalable particulate (see Appendix 1), which can be used as a preliminary screening method in combination with the results of the analysis of 'free' TGIC in the coating powders.

Requirements of the COSHH Regulations

3 The Control of Substances Hazardous to Health (COSHH) Regulations 1994⁴ are designed to ensure that the exposure of people at work to substances which could cause health damage is either prevented, or where that is not reasonably practicable, adequately controlled. Employers are required to make an assessment of the health risk created by such work, and to prevent or control exposure to the substances involved. The COSHH Regulations also require that persons who may be exposed to substances hazardous to health receive suitable and sufficient information, instruction and training. Employers must ensure that their responsibilities under the COSHH Regulations are fulfilled before allowing employees to undertake any procedure described in this MDHS.

Properties and uses

4 Triglycidyl isocyanurate (TGIC)⁵ is a solid which may

occur as a white opaque powder or granules or as clear crystals. It has low vapour pressure, low water solubility and no discernible odour. TGIC is primarily used as a cross-linking (curing) agent in polyester coating powders. It is also used in solder 'mask' inks in the printed circuit board industry.

Health effects

5 TGIC is toxic by inhalation and if swallowed. It is a severe eye irritant, and a mild skin and nasal irritant. Both pure TGIC, and TGIC-containing powder coatings have the potential to cause skin sensitisation in people which can lead to severe skin rashes. The effects of repeated long-term exposure to TGIC have not been well studied, but kidney damage has been observed in rats repeatedly exposed to TGIC by oral dosing, and damage to the developing sperm cells has been observed in mice repeatedly exposed to TGIC orally and by inhalation. There is animal evidence that exposure of males to TGIC produces genetic changes in the sperm which may lead to heritable effects in the offspring. There is cause for concern that these effects could occur in humans, so that TGIC has been classified in the EU as a Category 2 mutagen. The reproductive toxicity of TGIC has not been well studied. Information on the health effects of TGIC is published in HSE guidance Note EH64 (1997 supplement).⁵

6 TGIC has been assigned the following risk phrases under the Chemicals (Hazard Information and Packaging for Supply) Regulations 1994 as amended (CHIP Regulations).

R23/25	Toxic by inhalation and if swallowed
R41	Risk of serious damage to eyes
R43	May cause sensitisation by skin contact
R46	May cause heritable genetic damage
R48/22	Harmful: danger of serious damage to health by prolonged exposure if swallowed
R52/53	Harmful to aquatic organisms, may cause long-term adverse effects in aquatic environment

Exposure

7 Most exposure to TGIC occurs in the manufacture and use of coating powders.⁵ Potential exposure can occur during weighing, mixing, extrusion and milling during manufacture; spraying of articles by users and cleaning operations using brushes and compressed air.

Exposure limits

8 The latest edition of HSE Guidance Note EH40⁶ or its successor should be consulted for the occupational exposure limits applicable to airborne TGIC. The maximum exposure limit (MEL) is at present 0.1 mg m⁻³ for an 8 hour time-weighted average reference period for TGIC. The criteria on which it is based are published in the HSE Guidance Note EH64 (1997 supplement).⁵

Analytical methods

9 This is not an HSE 'reference' method in the strict analytical sense of the word as there are frequently several alternative methods for determining a particular analyte. With the exception of a few specific cases, where an exposure limit is linked to a specific analytical method (eg for rubber fume or asbestos), the use of methods not included in the MDHS series is acceptable provided they have the accuracy and reliability appropriate to the application.

10 This method has been validated to demonstrate that it is capable of meeting the stated performance parameters. If an alternative method is used it is necessary to determine and state performance parameters for that method.

11 A preliminary screening method is described in Appendix 1 for information.

PRINCIPLE

12 A measured volume of sample air is drawn through a silanised glass fibre filter⁷ mounted in an inhalable dust sampler. After sampling, the filter is extracted. The method used to extract the sample is dependent on whether TGIC is present in air alone or in a premix or coating powder (paragraphs 62 and 63). The two extraction methods have been optimised to extract either TGIC and premix or coating powder samples. The solution is analysed by High Performance Liquid Chromatograph (HPLC) with a UV detector and the amount of TGIC present in the sample quantified by comparison with a range of standard solutions.^{1,2} The concentration of TGIC is calculated from the volume of air sampled and the amount of TGIC found on the filter.

13 This analytical method is validated between 0.01 and 0.2 mg m⁻³ pure TGIC for sample volumes of 200 L.

Note 1: HSE Guidance Note EH 42⁸ advises employers about how they should conduct investigations into the nature, extent and control of exposure to substances hazardous to health which are present in workplace air. The object of air monitoring is usually to determine

worker exposure, and therefore the procedures described in this method are for personal sampling in the breathing zone. The method may also be used for background or fixed location monitoring. It cannot be used to measure instantaneous or short-term fluctuations in concentration.

Detection limits

14 The detection limit for TGIC is typically around 0.18 µg per sample. For a 200 L volume air sample this corresponds to a detection limit of 0.9 µg m⁻³.

Overall uncertainty

15 The overall uncertainty has been calculated using the following CEN definition⁹ for each batch of samples:

$$\text{Overall uncertainty} = |\text{Bias}| + 2 (\text{precision})$$

For this method the bias was calculated as the possible bias of the flow meter (2%) and the average difference between the stated and analysed concentration of a series of premixes of known concentration (2%). The precision was calculated using the pump precision (5%) and the average precision of the analysis of a series of premix and cured powders (8%). This gives an overall uncertainty of 23%.

16 The performance meets the European Committee for Standardisation (CEN) performance criteria for analytical methods.⁹ These state that the overall uncertainty in a determination should be less than or equal to 50% for results between half and twice the limit value. Further information on quality assurance can be found in MDHS 71.¹⁰

Interferences

17 Organic components which have the same or nearly the same retention time as the analyte of interest during the HPLC analysis will interfere. Interferences can be minimised by proper selection of HPLC conditions. Analysis of a sample of the bulk premix or coating powder before analysis of the samples is advised in order to adjust the HPLC conditions used if an interference is observed.

Stability

18 Premix and coating powders were stored over three weeks. The recovery did not change over this period or on exposure to humid air.

19 TGIC was spiked onto filters over the range 2 to 40 µg and stored for 1 week at 4°C. Over this range, the recovery after one day's storage was 100% with a standard deviation of 4%. At one week's storage, recovery at 20 to 40 µg was 94% with a standard deviation of 6%, but at 2 µg the recovery was 68% with a standard deviation of 11%. This does not meet the CEN requirements for storage¹¹ at 2 µg.

REAGENTS

20 During the analysis only use reagents of recognisable analytical reagent grade. Suitable personal protection (eg gloves and safety spectacles) should be

used when handling the reagents listed below. Use only distilled or de-ionised water, or water of equal purity. Do not pipette by mouth.

Silanising solution

21 5% dichlorodimethylsilane in cyclohexane.

Methanol

22 'HPLC' grade methanol.

Acetone

23 'HPLC' grade acetone.

Cyclohexane

Dichlorodimethylsilane

HPLC mobile phase

24 Dissolve 1.15 g of phosphoric acid in 1 L de-ionised water and adjust to pH6 with sodium hydroxide solution. Add 900 ml of this solution to 100 ml acetonitrile and mix thoroughly. De-gas by filtration under vacuum or with helium.

Tetrahydrofuran

25 'HPLC' grade tetrahydrofuran (THF).

Acetonitrile

26 'HPLC' grade acetonitrile.

Phosphoric acid

27 Phosphoric acid 85% w/v.

Sodium hydroxide solution

28 Sodium hydroxide solution in de-ionised water.

Laboratory detergent solution

29 A laboratory grade detergent suitable for cleaning of samplers and labware, diluted with water according to the manufacturer's instructions.

Helium

30 Compressed and regulated to around 5 psi for eluent degassing.

Nitrogen

31 Compressed and regulated to a flow suitable for drying samples.

SAMPLING EQUIPMENT

32 Major UK suppliers of sampling equipment are listed in MDHS 14/2.³

Sampling heads

33 Sampling heads suitable for use in this method are described in MDHS 14.³

Filters

34 Filters of a diameter suitable for use in the selected sampler (paragraph 33). The chosen filter type should be suitable for the collection and analysis of stable samples of TGIC, premix and cured powder coatings. Silanised glass fibre filters (paragraph 44) have been found to be suitable.

Sampling pumps

35 Sampling pumps will need to have an adjustable flow rate, incorporating a flow meter or flow fault indicator, capable of maintaining the appropriate flow rate (paragraph 44) to within 5% of the nominal value throughout the sampling period (paragraph 45), and capable of being worn by persons without impeding work activity.³ The pumps shall give a pulsation-free flow (if necessary, a pulsation damper shall be incorporated between the sampling head and the pump, as near to the pump as possible). Flow-stabilised pumps may be required to maintain the flow rate within the specified limits.

Flow meter

36 Flow meter, portable, capable of measuring the appropriate flow rate (see paragraph 44) to within $\pm 2\%$, and calibrated against a primary standard.³

Note 2: *The flow meter incorporated into the pump may be used provided that it has adequate sensitivity, that it has been calibrated against a primary standard with a loaded sampler in line, and that it is read in a vertical direction if it is the supported float type. However, it is important to ensure that there are no leaks in the sampling train between the sampling head and the flow meter, since in this event a flow meter in the pump or elsewhere in line will give an erroneous flow rate.*

Note 3: *A soap bubble flow meter may be used as a primary standard, provided its accuracy is traceable to national standards (see Appendix 2).*

Ancillary equipment

37 Flexible plastic tubing, of a diameter suitable for making a leakproof connection from the sampler to the sampling pump; belts or harnesses to which the sampling pump can conveniently be fixed, unless the pump is sufficiently small to fit in the worker's pocket; flat-tipped tweezers for loading and unloading the filters into samplers; and filter transport cassettes or similar, if required (see paragraph 55), to transport samples to the laboratory and filters for filtering aqueous samples before HPLC analysis.

LABORATORY APPARATUS

Glassware

38 A selection of laboratory glassware, including pipettes, beakers, measuring cylinders and volumetric flasks, class A complying with the requirements of BS 1792.¹²

Micropipettes

39 A set of adjustable positive adjustment micropipettes, calibrated against a primary standard, for preparation of calibration and sample solutions.¹³

Balance

40 A balance, calibrated against a primary standard, for the preparation of the calibration standards. The balance should be capable of weighing to ± 0.1 mg over the range 0 to 100 g.

Miscellaneous

41 Other equipment needed for this analysis is:

- vacuum filtration unit suitable for de-gassing HPLC solvents;
- vortex mixer;
- ultrasonic bath;
- nitrogen supply for drying samples;
- 100 μ l syringe.

High performance liquid chromatograph

42 An HPLC with UV detector is suitable. However, using a diode array detector improves the accuracy of the method by enabling purity and spectra to be confirmed. The sensitivity of this method is improved if temperature fluctuations are avoided, for example by thermostating the HPLC column.

Disposable gloves

43 Disposable gloves, impermeable, to avoid the possibility of contamination from the hands and to protect them from harmful substances. PVC gloves are suitable.

SAMPLING

Sampling procedure

44 Silanised glass filters⁷ are used (paragraph 46) in a sampling head suitable for sampling inhalable dust (MDHS 14³ describes suitable samplers). A suitable sample volume is 200 L. Increasing this sample volume will improve the detection limit for the method.

45 For long-term samples, select a sampling period of an appropriate duration, such that the filter does not become overloaded with particulate material (note that an 8-hour time-weighted average concentration may be derived from the results of two or more consecutive samples, as described in Guidance Note EH 42).⁸

Preparation of sampling equipment

46 Glass fibre filters are silanised before use.⁷ The filters are immersed in a 5% solution of dichlorodimethylsilane in

cyclohexane for 5 minutes. The excess reagent is decanted and added to methanol in order to deactivate the reagent before disposal. The filters are rinsed in fresh cyclohexane twice to remove residual reagent. The filters are then rinsed in methanol and allowed to stand in fresh methanol for 5 minutes. The methanol is decanted and the filters rinsed in acetone. The filters are then rinsed in water in order to remove hydrochloric acid generated during silanisation. The filters are ready for use after drying in an oven.

47 Clean the sampling heads before use. Disassemble the samplers, soak them in laboratory detergent solution (paragraph 29), rinse thoroughly with water, wipe with absorptive tissue and allow to dry thoroughly before reassembly.

48 Load the filters (paragraph 46) into clean, dry sampling heads (paragraph 47) using clean flat-tipped tweezers (paragraph 37). Connect each loaded sampling head to a sampling pump using plastic tubing and ensuring that no leaks can occur. Switch on the pump, attach the calibrated flow meter (paragraph 36) to the sampling head so that it measures the flow through the sampler inlet orifice, and set the appropriate flow rate (paragraph 44) with an accuracy of $\pm 5\%$ of the nominal value through the sampling period.³ Switch off the pump and seal the sampler with a protective cover to prevent contamination during transport to the sampling position.

Collection of samples

49 Fix the sampling head to the worker, on their lapel and as close to the mouth and nose as possible.³ Then, either place the sampling pump in a convenient pocket or attach it to the worker in a manner that causes the minimum inconvenience, eg to a belt around the waist (paragraph 37). When ready to begin sampling, remove the protective cover from the sampling head and switch on the pump. Record the time at the start of the sampling period, and if the pump is equipped with an elapsed time indicator, ensure that this is set to zero.

50 Since it is possible for a filter to become clogged, monitor the performance of the sample periodically, a minimum of every two hours (or more frequently if heavy filter loadings are suspected). Terminate sampling and consider the sample to be invalid if the flow rate is not maintained to within $\pm 5\%$ of the nominal value throughout the sampling period.³

Note 4: *Regular observation of the flow fault indicator is an acceptable means of ensuring that the flow rate of flow-stabilised pumps is maintained satisfactorily, provided that the flow fault indicator indicates malfunction when the flow rate is outside $\pm 5\%$ of the nominal value.*

51 At the end of the sampling period, measure the flow rate with an accuracy of $\pm 5\%$ using the calibrated flow meter (paragraph 36), switch off the sampling pump, and record the flow rate and sampling time. Also observe the reading on the elapsed time indicator, where fitted, and consider the sample to be invalid if the reading on the elapsed time indicator and the timed interval between switching on and switching off the sampling pump do not

agree to within $\pm 5\%$ since this may suggest that the sampling pump has not been operating throughout the sampling period. Reseal the sampling head with its protective cover and disconnect it from the sampling pump.

52 Carefully record the sample identity and all relevant sampling data (see Appendix 3). Calculate the mean flow rate by averaging the flow rate measurements throughout the sampling period and calculate the volume of air sampled, in litres, by multiplying the flow rate in litres per minute by the sampling time in minutes.

53 With each batch of ten samples, submit for analysis at least two unused filters from the same lot of filters used for sample collection. Subject these filter blanks to the same handling procedure as the samples, but draw no air through them.

54 A portion (approximately 20 ml) of every cured powder coating and premix used in the factory during the period of sampling must also be retained for analysis.

Transportation

55 The filters are placed in sealed, labelled metal tins and the bulk samples in sealed containers. Samples are kept in the fridge and analysed within one week of sampling.

ANALYSIS

56 Wear disposable gloves (paragraph 43) to reduce the possibility of contamination and to protect hands from harmful solvents/reagents.

Cleaning of glassware

57 Before use, clean all glassware to remove any residual grease or chemicals. Firstly soak overnight in laboratory detergent solution (paragraph 29) and then rinse thoroughly with water.

Preparation of sample and blank solutions

58 The method used to extract the sample is dependent on the type of sample taken (paragraphs 62 and 63). These methods have been optimised to extract either TGIC and premix or coating powder samples.

Samples of bulk coatings

59 Analysis of either samples of the bulk premix (if available) or coating powder is recommended before desorption of the samples in order to modify the HPLC conditions if any interfering peaks are seen in the chromatograms. The amount of 'free' TGIC measured in the coating powder analysis may also be used in combination with sampling for total inhalable dust as a screening method (see Appendix 1).

60 Samples of premix bulk are ground up to provide representative portions for analysis and approximately 8 mg of the premix powder is weighed out into six vials containing a silanised glass fibre filter and extracted (paragraph 62).

61 Coating powders are supplied as a fine powder. Approximately 8 mg of the coating powder is weighed out into six vials containing a silanised glass fibre filter and extracted (paragraph 63).

Samples of TGIC and premix in air

62 The sample filters are wetted with 100 μ l acetonitrile and 2 ml mobile phase added. They are sonicated for one hour and the solution filtered before HPLC analysis.

Samples of TGIC and coating powders in air

63 The sample filter is placed in a vial containing 2 ml THF and mixed on a vortex mixer to dissolve the polyester coating and TGIC. Water (0.5 ml) is added to the vial to precipitate the polyester coating and the mixture thoroughly mixed on the vortex mixer. This is repeated for a second portion of water. The sample is dried under nitrogen and a portion of acetonitrile (2 ml) added to the sample. The sample is sonicated in acetonitrile for 30 minutes and a portion (1 ml) transferred to a volumetric flask. The solution is dried under nitrogen and HPLC mobile phase (1 ml) is added to the sample before sonication for 30 minutes. The solution is filtered and analysed by HPLC.

Blank samples

64 Blank samples are subjected to the same analytical procedure as the other samples in the set (paragraph 62 and 63).

Preparation of calibration standards

65 At least six standard solutions of TGIC over the range 0.5-40 μ g/ml should be prepared daily by dissolving a known weight of TGIC in mobile phase and diluting as appropriate.

Chromatography

66 HPLC conditions that have been found suitable for TGIC are:

Column dimensions	100 mm x 4.5 mm ID with a 1 cm guard column
Column packing	S3 ODS2
Column temperature	20°C
Mobile phase	0.01 M phosphate buffer pH 6.0 in 10% acetonitrile
Flow rate	1 ml/min
UV detector	205 nm

The retention time of TGIC under these conditions is 10.3 minutes.

System calibration

67 Inject into the liquid chromatograph a known fixed volume of each standard solution (paragraph 65), eg 25 μ L. A standardised injection technique should be used such that reproducible areas are obtained. Prepare a calibration graph of peak response against analyte

concentration. The precision quoted in paragraphs 12-14 has been obtained using an autosampler.

Samples

68 Inject into the liquid chromatograph the same fixed volume of the solution from the desorbed sample of the bulk premix/coating powder. Read from the calibration graph the concentration of the analyte in the desorbed premix/coating powder. Analyse the blank in the same way.

69 The chromatogram is checked for interfering peaks and the recovery of 'free' TGIC calculated in the samples of the bulk materials. If necessary, the HPLC conditions are modified. Premixes are expected to contain only 'free' TGIC - however some TGIC will be cross-linked in the coating powder and the recovery of 'free' TGIC in coating powders will be less than the total amount added to the coating by the manufacturer (paragraph 1).

70 The sample filters are analysed as described in paragraph 68.

71 Where high TGIC concentrations are found, dilute the sample solutions with mobile phase to bring the concentration back within the calibration range (paragraph 65). Repeat the analysis and record the dilution factor.

CALCULATION OF RESULTS

'Free' or unbound TGIC in bulk samples

72 The percentage 'free' or unbound TGIC in the premix/coating can be calculated and used in the screening method (Appendix 1). The concentration of TGIC in the sample in $\mu\text{g ml}^{-1}$ is calculated by comparison with the calibration graph (paragraph 68). The free TGIC in the sample of the bulk material is then calculated from the following formula:

$$F = \frac{D(m - m_{\text{blank}})}{W} \times 100$$

where F = % free or unbound TGIC in the bulk by weight
 W = weight (μg) of bulk powder analysed
 m = concentration ($\mu\text{g/ml}$) of TGIC in sample
 m_{blank} = concentration ($\mu\text{g/ml}$) of TGIC in blank
 D = volume of desorbing solution (2.1 ml for pure TGIC and premix samples, 2 ml for powder coatings)

Volume of air sample

73 Calculate the volume, V, in litres, of each air sample (paragraph 52).

Mass concentration of analyte in air samples

74 Calculate the concentration of TGIC on the unextracted sample filter in $\mu\text{g ml}^{-1}$ by comparison with

the calibration graph (paragraph 68). Use the following formula to calculate the concentration of TGIC in mg m^{-3} .

$$T = \frac{D(m - m_{\text{blank}})}{V}$$

where T = TGIC concentration (mg m^{-3})
 V = sample volume (L)

Overall uncertainty

75 The overall uncertainty is calculated to be 23% of the result (paragraph 14).

TEST REPORT

76 Appendix 3 gives recommendations for information to be included in the test report.

APPENDIX 1

Gravimetric screening method

A gravimetric screening method can be used as a preliminary screening method for TGIC.

Sampling is carried out according to MDHS 14/2³ for total inhalable particulate. All the dust generated in the sampling area is assumed to come from 'free' TGIC (alone or in premix) or cured coating powder. The total air concentration of particulate is calculated and used to estimate exposure to TGIC.

For air samples containing coating powder only either the proportion of TGIC added to the coating by the manufacturer or the results of an analysis of a sample of the bulk coating powder (paragraph 71) can be used to estimate the proportion of TGIC in the particulate. For example, it is assumed that for a coating containing 4% by weight TGIC, 4% of the total particulate measured is assumed to be TGIC. As the amount of TGIC added to the coating by the manufacturer includes both 'free' and cross-linked TGIC, a more accurate estimate of exposure would be obtained using the results of an analysis of the bulk coating powder, ie the proportion of 'free' TGIC only in the powder (paragraph 72).

APPENDIX 2

Primary standard for calibration of a flow meter

The primary standard should preferably be a flow meter whose accuracy is traceable to national standards, used with careful attention to the conditions of the calibration certificate. A bubble flow meter may also be used. This is an arrangement whereby the pump under test draws a soap film up a calibrated tube. The passage of the film is accurately timed between two marks whose separation defines a known volume. The volume between the marks can be checked by filling the burette with water, allowing the temperatures to stabilise, drawing off a known volume and weighing the water, making allowance for the dependence of volume on temperature. A suitable bubble solution can be made by

mixing one part concentrated washing-up liquid, two parts glycerol and four parts water. The burette must be thoroughly wetted with the solution and several attempts at drawing the film up the tube may be necessary before the tube is wet enough for this to be achieved consistently. Traceability of the calibration will require checking of clocks and use of calibration weights.

APPENDIX 3

Recommendations for the test report

It is recommended that the test report should include the following information:

- (a) complete identification of the sample, including the date of sampling, the place of sampling, and the identity of the individual whose breathing zone was sampled;
- (b) reference to this MDHS and a description of any deviation from the procedures described;
- (c) the type and diameter of filter used, and the type of sampling head;
- (d) the type of sampling pump and flow meter used, the primary standard against which it was calibrated, and the range of flow rates for which the flow meter was calibrated;
- (e) the duration of the sampling time in minutes and/or the time at the start and at the end of the sampling period;
- (f) the volume of air sampled, in litres;
- (g) the name of the person who collected the sample;
- (h) the time-weighted average TGIC concentration found in the air sample, in milligrams per cubic meter and/or the mass collected on the filter, in milligrams;
- (i) the overall uncertainty of the method;
- (j) the name of the analyst;
- (k) the date of the analysis;
- (l) any unusual features noted during the determination.

ADVICE

Advice on this method can be obtained from the Health and Safety Executive, Health and Safety Laboratory, Broad Lane, Sheffield S3 7HQ (tel: 0114 2892000).

The Health and Safety Executive wishes, wherever possible, to improve the methods described in this series. Any comments that might lead to improvements would therefore be welcome and should be sent to the above address.

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| 43 | Styrene diffusive/thermal desorption/GC | | |
| 44 | Styrene diffusive/solvent desorption/GC | | |
| 45 | Ethylene dibromide solvent desorption/GC | | |
| 46 | Platinum AA | | |
| 47 | Rubber fume in air measured as total particulates and cyclohexane soluble material | | |
| 48 | Newspaper print rooms: measurements of total particulates and cyclohexane soluble material in air | | |

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