Canadian General Standards Board

Office des normes générales du Canada

CGSB

ONGC

164-GP-1MP
February/Février 1987

ICS 13.030.40

WITHDRAWAL

March 2004

CANADIAN GENERAL STANDARDS BOARD

LEACHATE EXTRACTION PROCEDURE

This CGSB document is hereby withdrawn due to its longevity as a provisional standard. There no longer exists the interest and support to revise the CGSB provisional document as a National Standard of Canada in light of the U.S. EPA Method 1311 — Toxicity Characteristic Leachate Procedure (TCLP). Contact the U.S. EPA for the most current version of the EPA Method 1311.

RETRAIT

Mars 2004

OFFICE DES NORMES GÉNÉRALES DU CANADA

MÉTHODE DE LIXIVIATION

Ce document de l'ONGC est retiré par le présent avis à cause de sa longévité en tant que norme provisoire. Il n'y a plus l'intérêt ni l'appui nécessaire pour réviser ce document provisoire de l'ONGC au niveau de Norme nationale du Canada compte tenu de la méthode américaine EPA Method 1311, Toxicity Characteristic Leachate Procedure (TCLP). Communiquer avec l'agence américaine (EPA) pour obtenir la dernière version de l'EPA Method 1311.

Canada

CORRIGENDUM

164-GP-1MP
February 1987

Leachate Extraction Procedure

ANNEX A (Page A1)

Please change the title of the annex to read: "ANNEX A TO CGSB 164-GP-1MP".

Withdrawn/Retirée

Canadian General Standards Board
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Leachate Extraction Procedure

This provisional standard sets out the requirements for determining the leachate extraction from solid waste materials

164-GP-1MP
February 1987

Canada

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The Secretary
Canadian General Standards Board
Ottawa, Canada
K1A 1G6

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Même si l'objet de la présente norme précise l'application première que l'on peut en faire, il faut cependant remarquer qu'il incombe à l'utilisateur, au tout premier chef, de décider si la norme peut servir aux fins qu'il envisage.

Plusieurs des tests requis aux termes des normes de l'ONGC sont dangereux. L'ONGC n'assume ni n'accepte aucune responsabilité pour les blessures ou les dommages qui pourraient survenir pendant les essais, peu importe l'endroit où ceux-ci sont effectués.

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Le Secrétaire
Office des normes générales du Canada
Ottawa, Canada
K1A 1G6

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Canadian General Standards Board
Office des normes générales du Canada

LEACHATE EXTRACTION PROCEDURE

This provisional standards has been published to meet an urgent requirement of the Ministry of Transport. It has not been approved by a technical committee nor ratified by the Minister's Advisory Council on CGSB.

It is intended that this standard will be formally approved and published as a bilingual National Standard of Canada in the near future.

Withdrawn/Retirée

164-GP-1MP
February 1987

PUBLISHED BY CANADIAN GENERAL
STANDARDS BOARD, OTTAWA, CANADA K1A 1G6

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PUBLIÉE PAR L'OFFICE DES NORMES GÉNÉRALES
DU CANADA, OTTAWA, CANADA K1A 1G6

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164-GP-1 MP

February 1987

CANADIAN GENERAL STANDARD BOARD

LEACHATE EXTRACTION PROCEDURE

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1. SCOPE

1.1 This standard sets out the requirements for determining the leachate extraction from solid waste materials

2. APPLICABLE PUBLICATIONS

The following publications are applicable to this standard:

2.1 United States Environmental Protection Agency

EPA SW-846 Test Methods For Evaluating Solid Waste, September 1986

2.2 American Society for Testing and Materials (ASTM)

D1193 Standard Specification for Reagent Water

2.3 Reference to the above publications is to the issues in effect as of the date of this standard, unless otherwise specified. For the source of these publications, see the Notes section.

3. APPARATUS

3.1 Sieve, 9.5 mm mesh opening, stainless steel or plastic material.

3.2 Stainless steel filtration unit, 142 mm diameter, minimum 1L capacity, capable of sustaining a pressure of 500 kPa, applied to the solution to be filtered.

3.3 Membrane filter, 142 mm diameter, 0.45 µm diameter pore size, made of synthetic organic material such as teflon, cellulose acetate, cellulose nitrate, nylon or polycarbonate and which is compatible with the leachate to be filtered. Teflon is recommended for organic constitutents.

3.4 Glass fibre prefilter, 124 mm diameter, 3 µm to 12 µm pore size range.

3.5 Vacuum filtration unit, 90 mm diameter.

3.6 Membrane filter 90 mm diameter, 0.45 µm diameter pore size, made of synthetic organic material such as teflon, cellulose acetate, cellulose nitrate, nylon or polycarbonate and which is compatible with the leachate to be filtered. Teflon is recommended for organic constitutents.

3.7 Glass fibre filter 70 mm diameter, 3 µm to 12 µm pore size range.

3.8 Solid waste rotary extractor - a device that rotates the bottles end over end about a central axis through 360 °, with a speed of 10 rpm. The dimensions of the box will depend on the needs of each laboratory. See Figure 1.

3.9 Structural Integrity Tester with a 31.8 mm diameter hammer weighing 0.33 kilogram and having a free fall of 152.4 mm. See Figure 2.

3.10 pH meter, with a readability of 0.01 pH unit and accuracy of ± 0.1 pH units.

3.11 Cylindrical bottles, wide mouth, 1250 mL capacity, polyethylene or glass with lined cap for inorganic constituents; glass with Teflon-lined cap or Teflon bottles for organic constituents.

4. REAGENTS and MATERIALS

4.1 Acetic acid 0.5 N. Dilute 29.4 mL of concentrated acetic acid [American Chemical Society (ACS) reagent grade] to 1000 mL with reagent water.

4.2 Reagent water, Type IV in accordance with ASTM Specification D 1193 "Reagent Water". For organic parameters, the reagent water shall be free of any organic substances to be analyzed , Type I in accordance with ASTM Specification D 1193.

4.3 Nitric acid, 10% (v/v). Add 100 mL of concentrated nitric acid (ACS reagent grade) to 900 mL of reagent water.

4.4 Nitrogen gas, pre-purified, scrubbed through a molecular sieve.

5. SAMPLING

5.1 Collect a sufficient amount of sample to provide approximately 100 g of solid material, using techniques set out in US EPA SW-846 "Test Methods For Evaluating Solid Waste".

Withdrawn/Retiree

6. PREPARATION of APPARATUS

6.1 All glassware and equipment that comes into contact with the sample shall be cleaned in the following way before each use.

6.2 Wash with a non-phosphate detergent solution.

6.3 Rinse twice with tap water.

6.4 Rinse twice with reagent water.

6.5 Wash with 10% nitric acid.

6.6 Rinse several times with reagent water.

6.7 Store bottles filled with 10% nitric acid, until ready to use.

6.8 Rinse several times with reagent water before use.

6.9 Rinse clean oven dried bottles with methylene chloride, followed by methanol, for organic constituents.

7. PROCEDURES

7.1 **Separation Procedure**

7.1.1 If the sample contains a distinct liquid and a solid phase, separate it into its component phases using the following procedure.

7.1.2 Determine the dry mass of the solids in the sample at 60°C, using a well homogenised sample. Use this mass to determine the amount of material to be filtered.

7.1.3 Assemble the filtration unit with a filter bed consisting of a 0.45 µm pore size membrane filter and a coarse glass fibre pre-filter upstream of the membrane filter (per manufacturer's instructions).

7.1.4 Select one or more blank filters from each batch of filters. Filter 50 mL portions of reagent water through each test filter and analyze the filtrate for the analytical parameters of interest. Note the volume required to reduce the blank values to acceptable levels.

7.1.5 Wash each filter used in the leach procedure with at least this predetermined volume of water. Filter under pressure until no water flows through the filtrate outlet.

7.1.6 Remove the moist filter bed from the filtration unit and determine its mass to the nearest ± 0.01 g.

Withdrawn/Retirée

7.1.7 Re-assemble the filtration unit, replacing the filter bed, as before.

7.1.8 Grind the sample with a mortar and pestle, to a size that will pass through the opening of the filtration unit (less than 9.5 mm).

7.1.9 Agitate the sample by hand and pour a representative fraction of the solid and liquid phases into the opening of the filtration unit. Filter a sufficient amount of the sample to provide at least 60 g of dry solid material.

7.1.10 Pressurize the reservoir very slowly with nitrogen gas by means of the regulating valve on the nitrogen gas cylinder, until liquid begins to flow freely from the filtrate outlet.

7.1.11 Increase the pressure step-wise in increments of 50 kPa to a maximum of 500 kPa, as the flow diminishes. Continue filtration until the liquid flow ceases or the pressurizing gas begins to exit from the filtrate outlet of the filter unit.

7.1.12 De-pressurize the filtration unit slowly using the release valve on the filtration unit. Remove and weigh the solid material together with the filter bed to ± 0.01 g. Record the mass of the solid material.

7.1.13 Measure and record the volume and pH of the liquid phase. Store the liquid at 4°C under nitrogen until required in 7.2.13.

7.1.14 Discard the solid portion if the mass is less than 0.5% (w/v) of the fraction taken. If not, carry out the extraction procedure in 7.2

Note: For mixtures containing coarse grained solids, where separation can be performed without imposing a 500 kPa differential pressure, a vacuum filtration unit with a filter bed as described in 7.1.3 may be used. Vacuum filtration must not be used if volatile organic compounds are to be analysed.

7.2 **Extraction Procedure**

7.2.1 Prepare a solid sample for extraction by crushing, cutting or grinding, to pass through a 9.5 mm mesh sieve. If the original sample contains both liquid and solid phases, use the solid material from 7.1.14. The structural integrity

Withdrawn/Retirée

procedure in 7.3 should be used for monolithic wastes which are expected to maintain their structural integrity in a landfill, (e.g. some slags and treated solidified wastes).

Note: Do not allow the solid waste material to dry prior to the extraction procedure.

7.2.2 Determine the moisture content of the de-watered sample, by drying a suitable fraction to constant weight at 60°C in an oven. Discard the dried solid material.

7.2.3 Place the equivalent of 50 g dry mass of the de-watered undried material into a 1250 mL wide mouth cylindrical bottle. Use additional bottles, if a larger volume of leachate is required for the analysis.

7.2.4 Add 800 mL (less the moisture content of the sample in mL) of reagent water to the bottle.

7.2.5 Cap the bottle and agitate it in the rotary extractor for 15 minutes before measuring the pH.

7.2.6 Measure and record the pH of the solution in the bottle using a pH meter, calibrated with buffers at pH 7.00 and pH 4.00. The solution should be stirred during the pH measurement.

7.2.7 If the pH is less than 5.2, carry out the procedure comencing at 7.2.10.1.

7.2.8 Add a sufficient volume of 0.5N acetic acid if the pH is greater than 5.2 to bring the pH to 5.0 ± 0.2.

Note: Maximum Amount of Acid: No more than 4 mL of 0.5N acetic acid per gram of dry mass of sample may be added during the entire procedure. If the pH is not lowered to 5.0 ± 0.2 with this amount, proceed with the extraction.

7.2.9 Cap the bottle and place it in the solid waste rotary extractor. Rotate the bottle and its contents at 10 rpm for 24 hours at room temperature (20°C to 25°C).

7.2.10 Monitor, and manually adjust the pH during the course of the extraction, if it is greater than 5.2. The following procedure should be carefully followed:

7.2.10.1 Measure the pH of the solution after 1 hour, 3 hours and 6 hours from the starting time. If the pH is above 5.2, reduce it to pH 5.0 ± 0.2 by addition of 0.5N acetic acid. If the pH is below 5.0 ± 0.2, do not make any adjustments.

Withdrawn/Retiree

7.2.10.2 Adjust the volume of the solution to 1000 mL with reagent water, if the pH is below 4.8 after 6 hours.

7.2.10.3 Measure and reduce the pH to 5.0 ± 0.2, if required, after 22 hours and continue the extraction for an additional 2 hours.

7.2.11 Add enough reagent water at the and of the extraction period so that the total volume of liquid is 1000 mL. Record the amount of acid added and the final pH of the solution.

7.2.12 Separate the material into its component liquid and solid phases as described under the Separation Procedure (7.1). Discard the solid portion.

Note: It may be necessary to centifuge the suspension at high speed before filtration, for leachates containing very fine grained particles.

7.2.13 Calculate the amount of free liquid from 7.1.13 corresponding to 50 g of the dry solid material. Add this amount to the leachate from 7.2.12.

Note: If the analysis is not performed immediately, store separate fractions of the leachate at 4°C, after adding appropriate preservatives for the analytical parameters of interest (See "US EPA SW-846 "Test Methods For Evaluating Solid Waste".

7.2.14 Analyze the combined solutions from 7.2.13 using methods set ot in US EPA SW-846 "Test Methods For Evaluating Solid Waste"and which are capable of detecting the contaminants listed in Annex A, that are likely to be present at the detection limits specified for each contaminant.

7.2.15 Report concentrations of contaminants in the combined leachate and the free liquid solution as mg/L.

7.2.16 Carry a blank sample through the entire procedure, using dilute acetic acid at pH 5.0 ± 0.2.

7.3 **Structural Integrity Procedure**

7.3.1 This procedure may be required prior to extraction for some samples as indicated in 7.2.1. It may be omitted for wastes with known high structural integrity.

7.3.2 Fill the sample holder with the material to be tested. If the sample of the waste is a large monolithic block, cut a portion from the block measuring 33 mm in diameter by 71 mm in length. For a treated waste (e.g. solidified waste) samples may be cast in a form with the above dimensions for

Withdrawn/Retirée

the purposes of conducting this test. In such cases, the waste should be allowed to cure for 30 days prior to further testing.

7.3.3 Place the sample holder in the structural integrity tester, then raise the hammer to its maximum height and allow it to fall. Repeat this procedure 14 times.

7.3.4 Remove the material from the sample holder, and perform the extraction procedure (7.2). If the sample has not disintegrated, it may be sectioned; alternatively use the entire sample (after weighing) and a sufficiently large bottle as the extraction vessel. The volume of reagent water to be initially added is 16 mL/g of dry sample mass. The maximum amount of 0.5N acetic acid to be added is 4 mL/g of dry sample mass. The final volume of the leachate should be 20 mL/g of dry sample mass.

8. NOTES

8.1 ASTM publications are available from The American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103.

8.2 Unied States Government puublications are available from the Superintendant of Documens, US Government Printing Office, Washington DC 20402.

Withdrawn/Retired

ANNEX A TO CGSB XX-GP-XXX

Contaminant	Concentration (mg/L)
2,4,5-TP / Silvex / 2-(2,4,5-Trichlorophenoxy) propionic acid	0.1
2,4-D	1.0
Aldrin + Dieldrin	0.007
Arsenic	0.5
Barium	10.0
Boron	50.0
Cadmium	0.05
Carbaryl / 1-Naphthyl-N-methyl carbamate / Sevin	0.7
Chlordane	0.07
Chromium	0.5
Cyanide	2.0
DDT	0.3
Diazinon / Phosphordithioic acid, o,o-diethyl o-(2-isopropyl-6-methyl-4-pyrimidinyl) ester	0.002
Endrin	0.002
Flouride	15.0
Heptachlor + Heptachlor epoxide	0.03
Lead	0.5
Lindane	0.04
Mercury	0.01
Methoxychlor / 1,1,1-Trichloro-2, 2-bis(p-methoxyphenyl) ethane	1.0
Methyl parathion	0.07

Withdrawn/Retiree

Contaminant	Concentration (mg/L)
Nitrate + Nitrite	100.0
Nitrilotriacetic acid	0.5
Nitrite	10.0
PCBs	0.03
Parathion	0.35
Selenium	0.1
Silver	0.5
Toxaphene	0.05
Trihalomethanes	3.5
Uranium	0.2

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