INTERNATIONAL

Designation: E 2242 – 02

Standard Test Method for Column Percolation Extraction of Mine Rock by the Meteoric Water Mobility Procedure[1]

This standard is issued under the fixed designation E 2242; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method provides a procedure for the column percolation extraction of mine rock in order to determine the potential for dissolution and mobility of certain constituents by meteoric water.

1.2 This test method is intended to describe the procedure for performing column percolation extractions only. It does not describe all types of sampling and analytical requirements that may be associated with its application.

1.3 The values stated in SI units are to be regarded as the standard.

1.4 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*

D 1193 Specification for Reagent Water[2]

D 1293 Test Methods for pH of Water[2]

D 6234 Test Method for Shake Extraction of Mining Waste by the Synthetic Precipitation Leaching Procedure[3]

E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals[4]

E 135 Terminology Relating to Analytical Chemistry for Metals, Ores and Related Materials[4]

E 389 Method for Particle Size or Screen Analysis at No. 4 (4.75-mm) Sieve and Coarser for Metal-Bearing Ores and Related Materials[5]

E 691 Practice for Conducting an Interlaboratory to Determine the Precision of a Test Method[6]

E 877 Practice for Sampling and Analysis of Iron Ores and Related Materials[5]

E 882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory[5]

E 1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method[5]

2.2 *Other References:*

Meteoric Water Mobility Procedure, Bureau of Mining Regulation and Reclamation, Nevada Division of Environmental Protection, 9/19/90

Standard Methods for the Examination of Water and Wastewater, 18th edition, APHA/AWWA/WEF, 1992

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, see Terminology E 135.

3.1.1 *meteoric water*—rainfall that has the potential to contact materials and mobilize soluble constituents from mine rock at a mining or mineral processing site.

3.1.2 *mine rock*—ore, waste rock or overburden excavated in order to recover metals or minerals during mining operations or coarse processed ore such as heap leach spoils.

4. Summary of Test Method

4.1 The test material is placed in a plastic column and contacted with an equal weight of water during a 24 h leaching period. The extract is collected and preserved for analysis of inorganic constituents.

5. Significance and Use

5.1 This test method is intended as a means for obtaining an extract from mine rock samples. The extract may be used to estimate the final pH and release of certain constituents of the test sample under the laboratory conditions described in this test method.

5.2 The pH of the extraction fluid used in this test method should reflect the pH of precipitation in the geographic region in which the mine rock is being evaluated.

5.3 This test method is designed to mobilize potential contaminants present in the solids, so that the resulting extract can be used to assess leachate that could potentially be produced from mine rock in the field.

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[1] This test method is under the jurisdiction of ASTM Committee E01 on Analytical Chemistry of Metals, Ores and Related Materials and is the direct responsibility of Subcommittee E01.02 on Ores, Concentrates, and Related Metallurgical Materials.

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[2] *Annual Book of ASTM Standards*, Vol 11.01.

[3] *Annual Book of ASTM Standards*, Vol 11.04.

[4] *Annual Book of ASTM Standards*, Vol 03.05.

[5] *Annual Book of ASTM Standards*, Vol 03.06.

[6] *Annual Book of ASTM Standards*, Vol 14.02.

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5.4 This test method has not been demonstrated to simulate actual site leaching conditions.

5.5 This test method produces extracts that are amenable to the determination of both major and minor (trace) constituents. When minor constituents are being determined, it is especially important that precautions be taken in sample preservation, storage and handling to avoid possible contamination of the extracts.

5.6 This test method is a comparative method intended for use as a routine method for monitoring mine rock. It is assumed that all who use this method will be trained analysts capable of performing skillfully and safely. It is expected that work will be performed in a properly equipped laboratory under appropriate quality control practices such as those described in Guide E 882.

6. Interferences

6.1 This test method may not be suitable for obtaining extracts from finely divided solids (such as: finely crushed drill cuttings, clays, sludges, mill tailings, etc.), due to difficulty in obtaining a representative extract solution. If it takes more than 48 h for the column to produce an extract which has a mass equivalent to 70 % of the dry test sample weight, use of an alternate extraction procedure may be required, such as Method D 6234.

7. Apparatus

7.1 *Drying Pans or Dishes*, for moisture content determinations, 1 kg capacity.

7.2 *Extraction Column*, PVC column of 15-cm (6-in.) O.D. of sufficient height to contain a minimum of 5 kg of test sample with a feed particle size that just passes a 5-cm (2-in.) sieve and sufficient additional height to contain applied water volume should poor percolation occur. For a 5 kg test sample, a 15 cm O.D. × 45 cm high column is recommended. Additional column height will be required for test sample quantities greater than 5 kg. The bottom of the column must be sealed and a solution discharge outlet situated above the sealed bottom of the column and below a perforated support plate. A drawing of a suitable extraction column is shown in Fig. 1.

7.3 *Filtration Device*, of a composition suitable to the nature of the analyses to be performed and equipped with a 0.45 µm pore size filter. An assembly for pre-filtration or a centrifuge may be required if 0.45 µm filtration is difficult.

7.4 *Filter Media*, glass wool (inert) or a plastic fiber pad is placed onto the support plate before loading the laboratory sample into the column, to minimize fines migration, and onto the top of the test sample after column loading, to aid in making an even water distribution.

7.5 *Laboratory Balance*, capable of weighing to 1.0 g.

7.6 *pH Meter*, with a readability of 0.01 units and an accuracy of at least ±0.05 units at 25°C.

7.7 *Tubing*, surgical or synthetic tubing sufficient in diameter and length for the extraction column assembly (pump, column outlet).

7.8 *Water and Extract Containers*, sufficient in size to contain the water added during extraction. Containers must be covered to avoid contamination.

7.9 *Water Metering Device*, a metering pump or constant head device to insure constant rate extraction fluid application during column percolation.

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[7] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean Type II reagent water at 18 to 27°C conforming to Specification D 1193.

9. Hazards

9.1 For precautions to be observed in the use of analytical methods associated with this standard, refer to Practices E 50.

10. Sampling and Sample Preparation

10.1 The amount of gross sample to be sent to the laboratory should he sufficient to perform the solids content determination as specified in 10.3, and to provide at least 5 kg of test sample on a dry weight basis for extraction. Gross sample weights in the range of 7 to 25 kg are appropriate.

NOTE 1—Information on obtaining representative samples can also be found in Pierre Gy's Sampling Theory and Sampling Practice[8] and in Practice E 877.

10.2 In order to prevent sample contamination or constituent loss prior to extraction, keep the samples in closed containers appropriate to sample type and desired analysis.

10.3 *Moisture Determination*—Remove the gross sample from the container and blend by coning or rolling to obtain a sample for feed moisture content with a minimum of 500 g dry weight.

10.3.1 Weigh the moisture test sample and dry to constant weight (± 0.05 %) at 105 ± 2°C and record the weight.

10.3.2 Calculate the moisture content of the test sample as follows:

$$M = \frac{100(B - A)}{B} \tag{1}$$

[redacted]

FIG. 1 Column Percolation Extraction Device (Meteoric Water Mobility Procedure)

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[7] *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington. DC. For suggestions on the testing of reagents not listed by the American Chemical Society, see *Analar Standards for Laboratory Chemicals*, BDH Ltd., Poole, Dorset, U.K., and the *United States Pharmacopeia and National Formulary*, U.S. Pharmaceutical Convention, Inc. (USPC), Rockville, MD.

[8] Pitard, F., *Pierre Gy's Sampling Theory and Sampling Practice*, Vols. I and II, CRC Press, 1989.

where:
A = mass of sample after drying, g,
B = wet mass of sample, g, and
M = moisture content, %.

10.4 Separate the remaining laboratory sample on a 5-cm (2-in.) sieve. Save the minus 5-cm fraction for recombination with the crushed plus 5-cm fraction. The screening time or end point is when additional periods of shaking fail to change the results by more than 0.3 % (Method E 389). For highly friable material an acceptable end point shall be determined experimentally.

10.5 Weigh the plus and minus 5-cm sieve fractions, calculate and record the weight percent retained on the 5-cm sieve as follows:

$$R = \frac{100C}{(C + D)} \quad (2)$$

where:
R = weight retained on the 5-cm sieve, %,
C = mass retained on the 5-cm sieve, g, and
D = mass passing the 5-cm sieve, g.

10.6 Crush or hand break the materials retained on the 5-cm sieve so that they just pass through the 5-cm (2-in.) sieve, combine with the saved fraction passed through the 5-cm sieve and mix the prepared sample.

10.7 Thoroughly blend the prepared sample and calculate the minimum test sample weight containing 5 kg dry weight, based on the feed moisture content from 10.3, as follows:

$$S = \frac{5 \times 10^5}{(100 - M)} \quad (3)$$

where:
S = minimum sample mass for testing, g and
M = moisture content, %.

10.8 Divide the prepared sample, if necessary, to obtain a test sample suitable for processing in the column apparatus but not less than the minimum sample weight from 10.7. and weigh to ± 1 g. Label and reserve any excess prepared sample for any additional testing which may be required.

10.9 Calculate the dry weight of the test sample as follows:

$$L = \frac{E(100 - M)}{100} \quad (4)$$

where:
L = dry mass of the test sample, g,
E = gross mass of the test sample, g, and
M = moisture content of the gross sample, %.

11. Procedure

11.1 Place filter media (7.4) in the bottom of the extraction column and load the laboratory sample incrementally (~1-kg/increment) into the column. To minimize particle segregation and compaction during column loading, the sample portions shall be dropped from no more than 0.6 m (24 in.) when introduced from the top of the column, and no tamping, shaking, or other methods to compact the sample will be employed. Place filter media over the test sample in the column.

11.2 Use a water addition rate of 3.5 mL/min for minimum weight samples in the range of 5 to 5.5 kg dry weight.

11.2.1 For samples greater than 5.5 kg dry weight, calculate the rate of water addition to the column to pass a weight of water equal to the dry weight of the test sample during the 24 h test period as follows:

$$F = \frac{L}{1440} \quad (5)$$

where:
F = water addition rate, mL/min, and
L = dry mass of the laboratory sample, g.

11.3 Measure and record the initial temperature and pH of the leaching water in accordance with Test Methods D 1293. Record the time and date that water addition is started.

11.4 Add water to the column at the rate specified in 11.2 until the weight of the effluent is equal to the dry mass of the laboratory sample. If the water will not freely percolate through the column, abort the test and repeat the evaluation using an alternate testing procedure, such as Test Method D 6234.

11.5 Discontinue water addition and effluent collection when the effluent weight is equal to the dry weight of the test sample or 48 h has elapsed, which ever occurs first. If, after 48 h, the effluent weight is not at least 70 % of the dry weight of the test sample, abort the test and select an alternate testing method for the sample, such as Test Method D 6234.

11.6 Thoroughly mix the effluent and let the column effluent continue to drain into a waste container.

11.7 Immediately measure and record the final pH of the effluent in accordance with Test Methods D 1293. Record the date, time and final effluent weight.

11.8 *Extract*—Quantitatively transfer 2-L of effluent through a large glass funnel to a filtration device equipped with a 0.45 µm filter for analyses of dissolved constituents. Prefiltration using a coarse porosity glass filter and centrifuge separation procedures may be necessary prior to filtration for some difficult to filter effluents. Immediately measure and record the final pH of the extract in accordance with Test Methods D 1293. Record the final effluent and extract weights.

11.8.1 Retain a 250-mL portion of the extract for metals analysis with its pH adjusted to less than 2 with nitric acid.

11.8.2 Retain a 250-mL portion of the extract for nutrients analysis with its pH adjusted to less than 2 with sulfuric acid.

11.8.3 Retain a 1-L portion of the extract for cyanide analysis with its pH adjusted to between 12 and 12.5 with sodium hydroxide. Store in a dark bottle at 4 ± 2°C, if the cyanide analysis is not performed immediately.

11.8.4 Refrigerate the remaining extract at 4 ± 2°C for mineral analyses.

11.9 Analyze the unfiltered effluent for specific constituents or properties, or use for biological testing procedures as necessary.

11.10 Analyses should be performed using appropriate ASTM test methods. Where no appropriate ASTM test methods exist, other test methods may be used, such as Standard Methods for the Examination of Water and Wastewater, and record the methods used in the report. Whether visible phase separation during storage of the extract or effluent occurs or

TABLE 1 Statistical Information—Test Variables

Material	Number of Labs	Variable	Mean	Measurement Units
Mine Waste Rock	8	Time	27.5	Hours
Heap Leach Spoils	8	Time	26.7	Hours
Mine Waste Rock	8	Temperature	21.7	Degrees C
Heap Leach Spoils	8	Temperature	21.8	Degrees C
Mine Waste Rock	8	Volume	4.99	Liters
Heap Leach Spoils	8	Volume	5.05	Liters

not, appropriate mixing should be used to ensure the homogeneity of the extract and effluent prior to their use in such analyses or testing.

11.11 Allow the remaining column effluent to drain until at least two minutes elapses between drops of effluent from the test column. There should be no apparent free water phase in the column at the end of the draining period. Record any visual changes in the column residue, extract or effluent which occurred during the test period.

11.12 Remove the test residue from the column and blend by coning or rolling and obtain a sample of at least 500 g for residual moisture content. Determine the residual moisture as in 10.3 and record the results. Reserve the residue for any additional testing required.

12. Report

12.1 Report the following information:

12.1.1 Source of the gross sample, date of sampling, methods of sampling and sample preservation, storage conditions, handling procedures, and length of time between sample collection and extraction;

12.1.2 Moisture content (10.3), time and temperature used in the determination of the moisture content and results from any analyses performed on other splits of the laboratory sample;

12.1.3 Description of the gross sample, including its physical characteristics and sieve analysis results (10.5);

12.1.4 Weight of the test sample on a wet and dry basis;

12.1.5 The pH of the water used for the extraction;

12.1.6 Water application rate, temperature, extraction time, effluent pH and the final effluent weight;

12.1.7 Filter pore size used and filter composition;

12.1.8 Use of a centrifuge or pre-filter, the pre-filter pore size and composition;

12.1.9 Observations of changes in the test material or leaching solution (11.11);

12.1.10 The final weight and pH of the extract, and the results of specific analyses;

12.1.11 Moisture content of the residue, time and temperature used in the determination of the moisture content, and results of any analyses performed on the residue; and

12.1.12 Dates on which extraction was started and completed, preservation used for extract portions, and dates of analyses. A detailed laboratory worksheet that may be helpful in performing the test method is shown in Fig. 2.

13. Precision and Bias

13.1 *Precision*—Eight laboratories cooperated in testing this method and obtained the precision data summarized in Tables 1-3.[9] The interlaboratory testing was planned according to Practice E 1601, following Test Plan A for destructive test methods with single analyses for duplicate test materials, rather than three or more test portions with triplicate analyses, due to the decision to perform most of the analytical work in one central laboratory. The objective of the test was not to measure the precision of the analytical methods but to determine the precision of the column extraction procedure. The Practice E 691 computer program was used to calculate the statistics.

13.1.1 The analyses conducted immediately by the individual laboratories showed precision in the quantitative range with the exception of acidity and dissolved solids for the mine waste rock, which exceeded the quantitative limit for R of 50 %. Many of the solution analyses conducted by the central laboratory could not be used for statistical purposes, since they were reported to be below detection. The precision of many analyses in the central laboratory also were not within the quantitative range even at relatively high concentrations, so the user of this method is cautioned to interpret the analyte concentrations in the extracts as a qualitative, not quantitative indication of the presence of those analytes at the concentrations measured. The heterogeneity and particle size distribution of the test samples may strongly influence the precision of this test method.

13.2 *Bias*—No information on the accuracy of this test method is known because suitable reference materials were not available for testing during the interlaboratory study. The user of this method is encouraged to employ accepted reference materials, if available, to determine the accuracy of this method as applied to a specific laboratory.

14. Keywords

14.1 column percolation; meteoric water mobility; mine rock

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[9] Supporting data have been filed at ASTM International Headquarters and may be obtained by requesting Research Report RR: E01-XXXX.

TABLE 2 Statistical Information—Individual Laboratory Analyses

Material	Number of Labs	Analyte	Mean	Measurement Units	Repeatability Standard Deviation (s_r, E 691)	Reproducibility Standard Deviation (s_R, E 691)	Repeatability (r, E 691)	Reproducibility (R, E 691)	%R
Mine Waste Rock	8	Moisture	18.0	% H_2O	0.691	1.31	1.93	3.68	20
Heap Leach Spoils	8	Moisture	11.7	% H_2O	0.388	1.11	1.09	3.11	27
Mine Waste Rock	8	Water pH	5.61	pH	Not Applicable	Not Applicable			
Heap Leach Spoils	8	Water pH	5.61	pH	Not Applicable	Not Applicable			
Mine Waste Rock	8	Effluent pH	2.79	pH	0.0561	0.122	0.157	0.341	12
Heap Leach Spoils	8	Effluent pH	8.43	pH	0.101	0.265	0.282	0.741	9
Mine Waste Rock	8	Extract pH	2.81	pH	0.074	0.142	0.206	0.398	14
Heap Leach Spoils	8	Extract pH	8.37	pH	0.101	0.368	0.284	1.030	12
Mine Waste Rock	8	Acidity	506	µg/g	44.5	115	125	321	63^A
Heap Leach Spoils	8	Alkalinity	81.4	µg/g	10.5	12.4	29.3	34.7	43
Mine Waste Rock	8	Dissolved Solids	1130	µg/g	175	262	490	734	65^A
Heap Leach Spoils	8	Dissolved Solids	671	µg/g	69.9	79.8	196	224	33

^{A} Not Quantitative.

TABLE 3 Statistical Information—Central Laboratory Analyses

Material	Number of Labs	Analyte	Mean	Measurement Units	Repeatability Standard Deviation (s_r, E 691)	Reproducibility Standard Deviation (s_R, E 691)	Repeatability (r, E 691)	Reproducibility (R, E 691)	%R
Mine Waste Rock	8	Aluminum	43.0	µg/g	3.30	6.83	9.23	19.1	44
Heap Leach Spoils	8	Aluminum	0.0858	µg/g	0.0464	0.0800	0.13	0.221	258^A
Heap Leach Spoils	8	Arsenic	0.238	µg/g	0.0236	0.0306	0.0660	0.0856	36
Mine Waste Rock	8	Barium	0.0252	µg/g	0.00301	0.00758	0.00843	0.0212	84^A
Heap Leach Spoils	8	Barium	0.0406	µg/g	0.0178	0.0303	0.0500	0.0847	209^A
Heap Leach Spoils	8	Boron	0.323	µg/g	0.0177	0.0368	0.0495	0.103	32
Mine Waste Rock	8	Cobalt	0.0599	µg/g	0.0078	0.0105	0.0215	0.0295	49
Heap Leach Spoils	8	Cobalt	0.1020	µg/g	0.00921	0.0105	0.0258	0.0295	29
Mine Waste Rock	8	Chromium	0.0953	µg/g	0.00968	0.0193	0.0271	0.0541	57^A
Mine Waste Rock	8	Copper	2.38	µg/g	0.208	0.458	0.581	1.28	54^A
Mine Waste Rock	8	Iron	39.3	µg/g	3.31	9.47	9.26	26.53	68^A
Heap Leach Spoils	8	Iron	0.148	µg/g	0.0336	0.0401	0.0942	0.112	76^A
Mine Waste Rock	8	Potassium	5.06	µg/g	0.890	1.61	2.49	4.5	89^A
Heap Leach Spoils	8	Potassium	7.01	µg/g	0.281	0.470	0.786	1.32	19
Mine Waste Rock	8	Lithium	0.0804	µg/g	0.0074	0.0092	0.0207	0.0259	32
Heap Leach Spoils	8	Lithium	0.0495	µg/g	0.00218	0.00322	0.0061	0.00902	18
Mine Waste Rock	8	Magnesium	18.0	µg/g	1.37	2.67	3.83	7.49	42
Heap Leach Spoils	8	Magnesium	5.40	µg/g	0.542	0.542	1.52	1.52	28
Mine Waste Rock	8	Manganese	3.83	µg/g	0.333	0.589	0.931	1.65	43
Heap Leach Spoils	8	Molybdenum	0.0514	µg/g	0.00347	0.0057	0.0097	0.0159	31
Mine Waste Rock	8	Sodium	5.62	µg/g	1.21	1.69	3.39	4.73	84^A
Heap Leach Spoils	8	Sodium	138	µg/g	7.50	10.7	21.0	29.8	22
Mine Waste Rock	8	Nickel	0.245	µg/g	0.0187	0.0400	0.0529	0.112	46
Mine Waste Rock	8	Lead	2.71	µg/g	0.117	0.260	0.328	0.727	27
Mine Waste Rock	8	Strontium	0.286	µg/g	0.0276	0.0367	0.0773	0.103	36
Heap Leach Spoils	8	Strontium	0.563	µg/g	0.0553	0.0553	0.155	0.155	28
Heap Leach Spoils	8	Vanadium	0.0388	µg/g	0.00261	0.00593	0.00731	0.0166	43
Mine Waste Rock	8	Zinc	21.8	µg/g	1.90	3.68	5.33	10.3	47

^{A} Not Quantitative.

[redacted]

FIG. 2 Laboratory Worksheet

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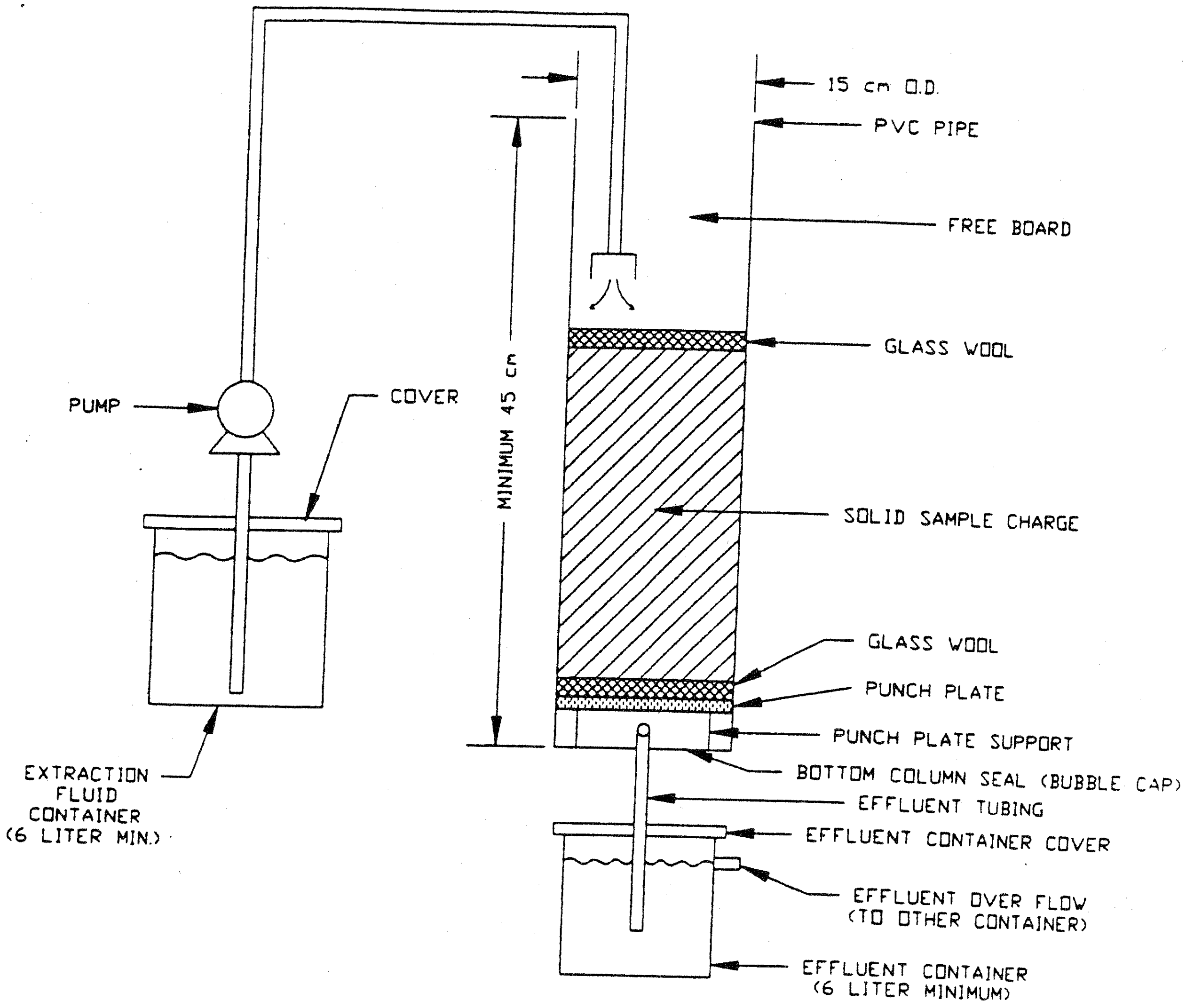
COLUMN PERCOLATION EXTRACTION DEVICE (METEORIC WATER MOBILITY PROCEDURE)



NOTES:

1) EXTRACTION DEVICE SET ON APPROPRIATE PLATFORM ABOVE EFFLUENT CONTAINER TO ALLOW GRAVITY FLOW OF EFFLUENT AND DRAIN.
2) CONSTANT INFLUENT APPLICATION RATE ACCOMPLISHED BY METERING PUMP OR CONSTANT HEAD APPARATUS.