Analytical Chemistry
Method No. 71-18
Job No. 1348006

GENERAL EXTRACTION PROCEDURE FOR RECOVERY OF ORGANIC TEST MATERIALS IN MIXED LIQUOR SAMPLES FROM SEMI-CONTINUOUS ACTIVATED SLUDGE UNITS

SCOPE

This procedure is intended for the extraction of various organic test materials from mixed liquor samples taken from semi-continuous activated sludge (SCAS) units for the purpose of measuring the rate of biodegradation of those materials.

PRINCIPLE

The method is based on the principle that most organic test materials can be extracted from SCAS mixed liquor samples with an appropriate organic solvent. The concentrated extract can then be measured for the test material by a suitable detection procedure.

REAGENTS AND APPARATUS

1. 25 ml. graduated cylinders
2. 250 ml. separatory funnels
3. 100 mm. 58° filtering funnel - Fisher Cat. No. 10-325E.
4. Kuderna-Danish evaporative concentrator - Ace Glass Cat. No. 6707 - 15 ml. graduated conical centrifuge tubes may be modified for use with this equipment.
5. Anhydrous sodium sulfate - Fisher Cat. No. S-421.
6. Solvent of choice (spectrograde or nanograde depending upon technique used for measurement).
7. Glass wool.
8. Carborundum boiling chips.
9. A steam heated water bath - a 16" diameter battery jar, 10" high, equipped with an overflow valve 1" from the top is filled with water and heated with steam for concentration of low boiling solvents in Kuderna-Danish evaporative concentrators.

PROCEDURE

1. Record the volume of the SCAS mixed liquor sample. (A description of the SCAS units and sampling instructions are found in Analytical Chemistry Method No. 71-32.)

2. After agitating, transfer the entire mixed liquor sample from the 25 ml. graduate cylinder to a 250 ml. separatory funnel.

3. Rinse the graduate cylinder several times with a total of 25 ml. of solvent, adding each solvent rinse to the separatory funnel.

4. Stopper the separatory funnel and shake vigorously for at least 1 minute. Allow the layers to separate and transfer the lower aqueous phase to a second separatory funnel.

5. Extract the aqueous layer a second time with a 25 ml. portion of hexane. After the layers have separated, add the first hexane extract to the second separatory funnel and transfer the aqueous layer to the original separatory funnel.

6. Repeat the extraction with a third 25 ml. portion of solvent. Discard the aqueous layer and combine the extracts.

7. Filter the combined extracts through a 4" funnel plugged with glass wool which is covered with sodium sulfate, washing with sufficient solvent to insure a quantitative transfer. Collect the filtrate in a Kunderna-Danish evaporative concentrator, add a small boiling chip, put the Snyder column in place, and reduce the solvent volume to less than the receiver volume by heating the apparatus in a 80-90°C. water bath. (CAUTION: SOLVENT VAPORS MUST BE VENTED TO A HOOD.)

8. Remove the receiver, dilute to the desired volume (or evaporate with a stream of nitrogen to reduce volume) for the measurement technique to be applied and cap tightly.

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Monsanto Company
Organic Chemicals Division
Applied Sciences Section
St. Louis, Missouri

DSW 312556

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