Method for Field Preparation and Analysis of Acrolein in Water

(Method AC 0689)

Sponsor:

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FIELD PREPARATION OF SAMPLES

After samples are removed from the canal the following preparation steps are to be followed.

1) The samples are to be filtered if needed.

2) A 25 ml portion of the sample is to be placed into a 2 oz. amber glass bottle labeled appropriately.

3) A 2 ml portion of PFPH/IS solution is to be added to each bottle.

4) The bottle is capped, shaken and placed in a cooler.

Preparation of standard curve based on a 25 ml sample size.

ppm Level	Stock Concentration ug/ml	mls of Stock
20	250	2
10	250	1
5	250	0.5
2	250	0.2
0.5	250	0.05
0.2	2.5	2
0.1	2.5	1
0.05	2.5	0.5
0.02	2.5	0.2
0.01	2.5	0.1

Preparations of control spikes based on a 25 ml sample size.

ppm Level	Stock Concentration ug/ml	mls of Stock
15	250	1.5
1.5	250	0.150
0.15	2.5	1.5
0.05	2.5	0.5
0.02	2.5	0.2

Preparation of internal standard and derivitizing mixture:

Pipet 10 ml of a 2.5 mg/ml solution of Crotonaldehyde into a 1000 ml volumetric containing about 900 ml of a 2.65 g/l PFPH solution. Then bring the volumetric to volume with the same PFPH solution.

Preparation of working stock standard solutions:

Prepare a mixture of 250 µg/ml of acrolein and 3-OH PFPH by pipeting 10 ml of a 500 µg/ml of each solution into a labeled container and mixing. This is the 250 µg/ml acrolein mixed standard.

The 2.5 µg/ml mixed standard is prepared by diluting the 250 µg/ml acrolein mixed standard at 1 ml to 100 ml then diluting with methanol.

EXTRACTION OF STANDARDS AND SAMPLES

Samples are extracted with ethyl acetate from the derivitized aqueous. The procedure used follows:

1) To each 125 ml separatory funnel a 5 ml portion of saturated NaCl is added.

2) Each sample is poured into the 125 ml separatory funnel. The sample container is rinsed with 25 ml of ethyl acetate and this is added to the 125 ml separatory funnel.

3) The funnel is shaken for 1 minute and the phases allowed to separate.

4) The lower aqueous phase is drained into the original sample container. The upper ethyl acetate layer is passed through a 3/4 - 1" layer of anhydrous Na_2SO_4 into a 25 X 200 mm screw top culture tube which has been calibrated to contain 50 ml.

5) The aqueous phase is returned to the appropriate separatory funnel. The sample container is again rinsed with 20 ml of ethyl acetate and this placed into the 125 ml separatory funnel.

6) The phases are allowed to separate and the lower aqueous layer is discarded. The ethyl acetate layer is passed through the same anhydrous Na_2SO_4 into the same screw top culture tube.

6.5) The anhydrous Na_2SO_4 is washed with two 5 ml portions of ethyl acetate.

7) The screw top culture tube is brought to the 50 ml volume mark and mixed.

8) The samples and standards are ready for injection onto the gas chromatograph.

8.5) All samples are detected at 1 ml of sample to 5 ml total volume with ethyl acetate.

GAS-LIQUID CHROMATOGRAPHY

The Acrolein and 3-Hydroxypropanaldehyde sample extracts were chromatographed using a Hewlett-Packard 5890 Gas Chromatrograph equipped with an electron capture detector with a Ni-63 source. Conditions employed for the assay utilized a DB-1701, 30 m X 0.32 mm capillary column. Approximate ranges of those conditions are listed below.

Column: J & W DB-1701, 30 m X 0.32 mm I.D., 1.5 µ film thickness.

Temperatures:

Injector: 200°C
Detector: 350°C
Column Oven: Initial: 120°C for 1 min.
Final: 220°C for 0 min., 280°C for 2 min.
Rate: 10°C, 30°C

Flow Rates:

Column: 15 psi Helium
Detector makeup: 60 ml/min P10-Argon/Methane

Precise records of instrument parameters for each analytical data set are found in the raw data.

DATA ACQUISITIONS AND CALCULATIONS

The Computer Automated Laboratory System (CALS®) allows for data acquisition, data analysis, results reporting and information management. Peak heights were measured using the CALS® system. Concentrations of each compound were then calculated using the CALS® system pre-programmed with a standard curve of ppm of each component versus peak height.

The resulting ppm values were taken directly from CALS® and then entered into Lotus® 2. Recoveries for field spikes for each component were then determined by the formula:

$$\% \text{ Recovery} = \frac{\text{ppm residue} - \text{ppm residue in control}}{\text{ppm residue added}}$$

The residues in the treated samples were not corrected for procedural recoveries.