

Sampling and Analytical Methods for Diacetyl and 2,3-Pentanedione

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Outline

- OSHA Method 1013 – Diacetyl
- OSHA Method 1016 – 2,3-Pentanedione
- OSHA Method 1012 – Diacetyl
- NIOSH Method 2557 – Diacetyl
- Correction of historical measurements obtained using NIOSH Method 2557

OSHA Method 1013 - Diacetyl

- Modification of OSHA Method PV2118
 - Greater sampling capacity
 - Avoids migration of diacetyl between sorbent beds during storage

OSHA Method 1013 - Diacetyl

- Sampling
 - 2 tubes containing 600 mg each dried silica gel
 - 0.05 L/min for 3 hr (9 L)
 - 0.2 L/min for 15 min (3 L)
 - Protect from light during and after sampling

OSHA Method 1013 - Diacetyl

- Analysis

- Extract tubes with 95% ethanol/5% water + 3-pentanone (internal standard)
- Gas Chromatography – Flame Ionization Detection
- Reliable Quantitation Limit:

$$0.37 \mu\text{g}/9\text{L air sample} = 41 \mu\text{g}/\text{m}^3 = 12 \text{ ppb}$$

OSHA Method 1016 – 2,3-Pentanedione

- Sampling
 - 2 tubes containing 600 mg each dried silica gel
 - 0.05 L/min for 3.33 hr (10 L)
 - 0.2 L/min for 15 min (3 L)
 - Protect from light during and after sampling
 - Ship samples cold and store refrigerated prior to analysis

OSHA Method 1016 – 2,3-Pentanedione

- Analysis
 - Extract tubes with 95% ethanol/5% water + 3-pentanone (internal standard)
 - Gas Chromatography – Flame Ionization Detection
 - Different conditions than Method 1013
 - Reliable Quantitation Limit:
 $0.38 \mu\text{g}/10\text{L air sample} = 38 \mu\text{g}/\text{m}^3 = 9.3 \text{ ppb}$
- Can sample and analyze for 2,3-pentanedione and diacetyl simultaneously (limited to 9L air sample)

OSHA Method 1012 - Diacetyl

- Sampling is identical to Method 1013
- Extract tubes with 95% ethanol/5% water + Pentafluorobenzylhydroxylamine hydrochloride (PFBHA) + 4-Bromobenzylbromide (internal standard)
- Gas Chromatography – Electron Capture Detection
- Reliable Quantitation Limit:
 $0.041 \mu\text{g}/9\text{L air sample} = 4.6 \mu\text{g}/\text{m}^3 = 1.3 \text{ ppb}$

OSHA Method 1012 - Diacetyl

- Can analyze samples after having analyzed by OSHA Method 1013, but will raise the Reliable Quantitation Limit by a factor of 2 (to 2.6 ppb)
- Derivatization procedure is not validated for 2,3-pentanedione

NIOSH Method 2557 - Diacetyl

- Most historical data on diacetyl exposures
- Sample with carbon molecular sieve (CMS) sorbent tube
- Extract with 99% acetone/1% methanol
- Gas Chromatography – Flame Ionization Detection
- Found to give poor recovery when sampling moderate to high humidity environments

Development of Correction Factor for NIOSH Method 2557 Measurements

- Publication by Cox-Ganser et al.
 - Journal of Occupational and Environmental Hygiene, 8:59-70 (2011).
- Generated known atmospheres of diacetyl at different concentrations and humidities and collected samples by Method 2557
- Found the bias in the measured value of diacetyl was affected by absolute humidity, length of storage before analysis , and actual concentration of diacetyl
- Developed a mathematical model that gives the corrected diacetyl concentration based on the measured concentration, the absolute humidity, and the storage time

Summary

- OSHA Methods 1013 and 1012 are validated methods to measure diacetyl in air and 1012 can measure below proposed REL of 5 ppb
- OSHA Method 1016 is a validated method to measure 2,3-pentanedione in air and the REL has been proposed to match the Reliable Quantitation Limit of 9.3 ppb
- A mathematical model has been developed to correct historical measurements of diacetyl by NIOSH Method 2557