

METHOD STATEMENT

Determinand:

Volatile Fatty Acids

(Acetic acid, propionic acid, n-butyric acid, isobutyric acid, n-valeric acid, isovaleric acid, n-caproic acid, isocaproic acid and methacrylic acid)

Matrix:

Sewage sludge, leachates and effluents

Principle of Method:

The sample is centrifuged, if necessary, and treated with formic acid to minimise adsorption effects on the gas chromatographic column (this also stabilises the liquor and prevents further biological activity, which would produce additional fatty acids). The individual acids present in the mixture are determined by gas chromatography with flame ionisation detection (GC-FID), using internal standardisation.

Sampling and Sample Preparation:

Samples are fixed with formic acid as soon as possible after receipt of the samples (sections **Error! Reference source not found.** to **Error! Reference source not found.**). If dilution is necessary, the fixed subsample is used, not the sample as received at the laboratory, as degradation will have taken place.

Samples are stable for 5 days (In-House Data) from sampling.

Interferences:

Any substance detected by the FID with the same retention time as an individual VFA will interfere.

Performance of Method:

Range of Application: 10mg/l to 500mg/l without dilution for acetic and propionic
10mg/l to 100mg/l without dilution for all other acids

Normal Reporting Level: 10mg/l as individual acids (5mg/l as carbon)
20mg/l as total volatile acids (10mg/l as carbon)

Determinand	LOD mg/l	Low Std*		High Std*	
		%Recovery	%RSD	%Recovery	%RSD
Acetic Acid	6.227	103.5	12.95	97.0	6.20
Propionic Acid	4.709	105.5	13.52	98.5	5.88
Isobutyric Acid	1.023	106.9	13.59	98.9	6.53
n-Butyric Acid	1.003	105.9	14.57	98.6	7.38
Isovaleric Acid	1.144	107.9	16.61	99.1	7.66
Methacrylic Acid	1.177	106.0	14.79	99.1	6.64
n-Valeric Acid	1.194	107.1	16.25	99.2	7.48
Isocaproic Acid	1.241	108.7	17.22	99.5	8.21
n-Caproic Acid	1.141	108.6	17.87	99.5	8.13

* Low Std: 20mg/l (100mg/l for acetic and propionic acids)
High Std: 80mg/l (400mg/l for acetic and propionic acids)



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Determinand		Final Effluent		Trade		Sewage Sludge		Groundwater	
		Low Spk	High Spk	Low Spk	High Spk	Low Spk	High Spk	Low Spk	High Spk
Acetic Acid	%Recovery	89.1	96.0	86.0	92.7	86.2	91.5	91.3	93.4
	%RSD	10.98	6.81	8.69	5.49	6.28	7.23	10.85	6.01
Propionic Acid	%Recovery	91.3	97.2	87.7	94.1	88.5	93.5	93.0	94.5
	%RSD	10.55	6.71	8.91	4.38	6.46	8.92	11.45	6.32
Isobutyric Acid	%Recovery	92.1	96.9	88.5	94.1	90.3	94.6	93.3	94.4
	%RSD	11.35	8.50	9.77	6.10	8.64	10.31	12.24	8.17
n-Butyric Acid	%Recovery	92.1	96.6	88.5	93.7	88.3	93.3	94.5	94.0
	%RSD	12.88	8.08	11.85	5.61	9.47	10.44	14.47	7.69
Isovaleric Acid	%Recovery	93.2	96.4	88.8	93.8	90.9	94.3	96.2	94.2
	%RSD	14.80	8.84	13.64	6.72	11.18	11.36	16.21	8.63
Methacrylic Acid	%Recovery	92.1	97.1	87.9	93.9	87.7	93.0	94.7	94.6
	%RSD	13.02	7.63	11.94	5.80	11.40	10.44	14.37	7.79
n-Valeric Acid	%Recovery	93.6	96.8	89.4	94.1	89.6	93.9	96.7	94.4
	%RSD	14.49	8.55	13.54	6.32	12.61	11.52	16.01	8.25
Isocaproic Acid	%Recovery	94.5	96.6	89.4	94.2	91.1	95.5	98.1	94.2
	%RSD	16.36	9.60	15.21	7.35	13.09	12.46	17.36	8.91
n-Caproic Acid	%Recovery	94.6	96.5	89.8	94.1	92.0	94.9	98.0	94.4
	%RSD	16.28	9.66	14.85	7.04	12.72	11.37	17.55	8.63

Determinand		Landfill Leachate		Soil Leachate		Surface Water	
		Low Spk	High Spk	Low Spk	High Spk	Low Spk	High Spk
Acetic Acid	%Recovery	90.0	95.4	89.4	94.4	91.0	94.2
	%RSD	10.15	7.14	10.80	3.15	8.50	6.95
Propionic Acid	%Recovery	92.3	97.6	92.0	96.0	92.3	95.7
	%RSD	8.55	9.92	10.28	2.75	8.54	6.43
Isobutyric Acid	%Recovery	93.0	98.2	93.0	96.3	461.7	478.4
	%RSD	9.54	11.59	9.93	3.61	8.54	6.43
n-Butyric Acid	%Recovery	92.5	97.5	92.6	95.5	93.5	95.3
	%RSD	10.37	11.73	13.18	3.49	12.11	7.31
Isovaleric Acid	%Recovery	94.1	98.2	94.0	96.0	95.0	95.5
	%RSD	12.72	13.25	14.52	4.83	13.65	7.81
Methacrylic Acid	%Recovery	93.0	97.8	93.0	96.2	94.0	96.0
	%RSD	11.27	11.51	13.38	3.90	11.22	6.80
n-Valeric Acid	%Recovery	93.9	98.2	94.6	96.1	95.4	95.7
	%RSD	12.82	12.66	14.01	4.52	13.55	7.45
Isocaproic Acid	%Recovery	95.4	98.5	95.3	96.2	96.7	95.6
	%RSD	14.29	14.01	15.40	5.57	14.95	8.67
n-Caproic Acid	%Recovery	95.2	98.6	95.8	96.3	96.7	95.4
	%RSD	14.22	14.03	15.08	5.61	14.98	8.38

References:

Determination of Volatile Fatty Acids in Sewage Sludge, 1979, HMSO. ISBN 0 11 751462 4. Method A

