

# Solutions for Sample Digestion in the Laboratory

## Inorganic Sample Preparation

The objective of sample preparation in inorganic analysis is to meet the requirements for a substantial, trouble free determination of the analyte. The most important general methods of sample preparation in any kind of matrix are:

1. Conversion of the sample into a form suitable for the determination (**dissolution**)
2. Destruction of the matrix (**digestion**)
3. Isolation of the analyte from interfering substances that may be present (**separation**)
4. Enrichment of the sample with respect to trace analytes (**concentration**)

The dissolution step is designed to compensate for inhomogenities in the sample. Dissolution and digestion also simplify the subsequent calibration step, ensuring that both the sample and the calibration solutions are in essentially the same chemical and physical state. The extent to which matrix constituents interfere in the determination process is significantly reduced by digestion, leading to a lower limit of detection for the determination. Digestion also facilitates concentration and separation steps.

**Wet sample preparation** is an essential part of chemical analysis and can thus be found in all kind of laboratories controlling the composition and quality of products for inorganic and organic compounds or contaminations

Most of the analytical methods, like titration, wet chemical analysis, or other types of instrumental analysis require liquid samples. Therefore all kind of samples must be dissolved or digested prior to analysis.

Wet digestion with oxidizing acids is the most common sample preparation procedure. This category can be extended to include processes involving bases or nonoxidizing acids as ashing reagents.

**Concentrated acids** with the requisite high degree of purity are commonly used.

To find the acid which is best suitable for digestion is a function of the sample matrix, the analyte, and the proposed determination method.

**Nitric acid** is an almost universal digestion reagent, since it does not interfere with most determinations.

However, nitric acid has a rather low boiling point (122 °C), and its oxidizing power is often insufficient under atmospheric-pressure conditions.

**Hydrogen peroxide** and **hydrochloric acid** can usefully be employed in conjunction with nitric acid as a way of improving the quality of a digestion. Hydrochloric acid and **sulfuric acid** may interfere with the determination of certain metals through the formation of stable compounds. Safety considerations are particularly important when using **perchloric acid**. Silicate samples require the further addition of **hydrofluoric acid**.

|  | Description  | VWR Cat. No.   | Packaging         | Size  |
|--|--|----------------|-------------------|-------|
| Acid Digestion   | Sulfuric acid 95–97% for analysis EMSURE® ISO  | EM1.00731.1000 | glass bottle      | 1L    |
|  | Acetic acid (glacial) 100% anhydrous for analysis EMSURE® ACS,ISO,Reag. Ph Eur   | EM1.00063.1000 | glass bottle      | 1L    |
|  | Hydrochloric acid fuming 37% for analysis EMSURE® ACS,ISO,Reag. Ph Eur   | EM1.00317.1000 | glass bottle      | 1L    |
|  | Nitric acid 65% for analysis EMSURE® ISO   | EM1.00456.1000 | glass bottle      | 1L    |
|  | Perchloric acid 70–72% for analysis EMSURE® ACS,ISO,Reag. Ph Eur   | EM1.00519.1001 | glass bottle      | 1L    |
|  | ortho-Phosphoric acid 85% for analysis EMSURE® ACS,ISO,Reag. Ph Eur  | EM1.00573.1000 | plastic bottle    | 1L    |
|  | Boric acid for analysis EMSURE® ACS,ISO,Reag. Ph Eur   | EM1.00165.0100 | plastic bottle    | 100g  |
|  | Formic acid 98–100% for analysis EMSURE® ACS,Reag. Ph Eur  | EM1.00264.1000 | glass bottle      | 1L    |
|  | Citric acid monohydrate for analysis EMSURE® ACS,ISO,Reag. Ph Eur  | EM1.00244.0500 | plastic bottle    | 500g  |
|  | Trichloroacetic acid for analysis EMSURE® ACS,Reag. Ph Eur   | EM1.00244.0500 | glass bottle      | 100g  |
|  | Hydrochloric acid 32% for analysis EMSURE®   | EM1.00319.1000 | glass bottle      | 1L    |
|  | Acetic acid 96% for analysis EMSURE®   | EM1.00062.1000 | glass bottle      | 1L    |
|  | Hydrofluoric acid 40% for analysis EMSURE® ISO,Reag. Ph Eur  | EM1.00338.0500 | plastic bottle    | 500ml |
|  | Hydrofluoric acid 48% for analysis EMSURE® ACS,ISO,Reag. Ph Eur  | EM1.00334.0500 | plastic bottle    | 500ml |
| Acid Digestion (For Instrumental Analysis Sample Preparation)  | Acetic acid (glacial) 100% Suprapur®   | EM1.00066.0250 | glass bottle      | 250ml |
|  | Ammonia solution 25 % Suprapur®  | EM1.05428.0250 | plastic bottle    | 250ml |
|  | Boric acid 99.9999 Suprapur®   | EM1.00765.0050 | plastic bottle    | 50g   |
|  | Formic acid 98–100% Suprapur®  | EM1.11670.0250 | glass bottle      | 250ml |
|  | Hydrobromic acid 47% Suprapur®   | EM1.00306.0250 | glass bottle      | 250ml |
|  | Hydrochloric acid 32% for analysis EMSURE®   | EM1.00319.1000 | glass bottle      | 1L    |
|  | Hydrofluoric acid 40% Suprapur®  | EM1.00319.1000 | glass bottle      | 1L    |
|  | Hydrogen peroxide 30% Suprapur®  | EM1.07298.0250 | plastic bottle    | 250ml |
|  | Nitric acid 65% Suprapur®  | EM1.00441.0250 | plastic bottle    | 250ml |
|  | Oxalic Acid dihydrate Suprapur®  | EM1.00489.0100 | plastic bottle    | 100g  |
|  | Perchloric acid 70% Suprapur®  | EM1.00517.0250 | glass bottle      | 250ml |
|  | Ortho-phosphoric acid 85% Suprapur®  | EM1.00552.0250 | plastic bottle    | 250ml |
|  | Sodium hydroxide solution 30% Suprapur®  | EM1.05589.0250 | plastic bottle    | 250ml |
|  | Sulfuric acid 96% Suprapur®  | EM1.00714.0250 | glass bottle      | 250ml |
|  | Hydrochloric acid 30% Ultrapur   | EM1.01514.0250 | plastic bottle    | 250ml |
|  | Hydrogen peroxide solution 31% Ultrapur  | EM1.06097.1000 | fibre carton      | 1L    |
|  | Nitric acid 60% Ultrapur   | EM1.01518.0250 | plastic bottle    | 250ml |
| Sulfuric acid 96% Ultrapur                                     | EM1.01516.0250   | plastic bottle | 250ml             |       |
| Water Ultrapur   | EM1.01262.0500   | plastic bottle | 500ml             |       |
| Alkaline Digestion – Hydroxides (Caustic Alkalis)              | Potassium hydroxide pellets for analysis EMSURE®   | EM1.05033.1000 | plastic bottle    | 1kg   |
|  | Potassium hydroxide pellets for analysis (max. 0.05% Na) EMSURE® ACS,Reag. Ph Eur  | EM1.05029.1000 | plastic bottle    | 1kg   |
|  | Potassium hydroxide pellets for analysis (max. 0.002% Na) EMSURE® ACS,ISO,Reag. Ph Eur   | EM1.05021.0250 | plastic bottle    | 250g  |
|  | Sodium hydroxide pellets for analysis EMSURE® ISO  | EM1.06498.0500 | plastic bottle    | 500g  |
| Alkaline Digestion – Hydroxides (Caustic Solutions)            | Sodium hydroxide pellets for analysis (max. 0.0002% K) EMSURE® ACS,Reag. Ph Eur  | EM1.06495.0250 | plastic bottle    | 250g  |
|  | Sodium hydroxide solution min. 10% (1.1) for analysis EMSURE®  | EM1.05588.1000 | plastic bottle    | 1L    |
|  | Sodium hydroxide solution 21% for analysis   | EM1.05593.9025 | plastic container | 25L   |
|  | Sodium hydroxide solution min. 27% (1.30) for analysis (for the determination of nitrogen) EMSURE®   | EM1.05591.2500 | plastic bottle    | 2.5L  |
|  | Sodium hydroxide solution about 32% (for the determination of nitrogen) for analysis EMSURE®   | EM1.05590.2500 | plastic bottle    | 2.5L  |
|  | Sodium hydroxide solution min. 45% for analysis EMSURE®  | EM1.11360.2500 | plastic bottle    | 2.5L  |
| Alkaline Digestion Carbonates                                  | Sodium hydroxide solution 50% for analysis EMSURE®   | EM1.58793.1000 | plastic bottle    | 1L    |
|  | Potassium hydroxide solution 47% for analysis EMSURE®  | EM1.05545.1000 | plastic bottle    | 1L    |
|  | Potassium carbonate for analysis EMSURE® ACS,ISO,Reag. Ph Eur  | EM1.04928.0500 | plastic bottle    | 500g  |
|  | Sodium carbonate anhydrous for analysis EMSURE® ISO  | EM1.05588.0500 | plastic bottle    | 500g  |
|  | Potassium carbonate/sodium carbonate GR for analysis   | EM1.06683.0500 | plastic bottle    | 500g  |
| Oxidizing Digestion Agents (Nitrates)                          | Calcium carbonate precipitated for analysis of silicates EMSURE®   | EM1.02067.0500 | plastic bottle    | 500g  |
|  | Potassium nitrate for analysis EMSURE® ISO,Reag. Ph Eur  | EM1.05063.1000 | plastic bottle    | 1kg   |
|  | Sodium nitrate for analysis EMSURE® ACS,ISO,Reag. Ph Eur   | EM1.06537.0500 | plastic bottle    | 500g  |
|  | Ammonium nitrate for analysis EMSURE® ACS  | EM1.01188.0500 | plastic bottle    | 500g  |
| Oxidizing Digestion Agents (Chlorates)                         | * Normally in combination with alkaline metal hydroxides or carbonates   |                |                   |       |
|  | Potassium chlorate for analysis EMSURE®  | EM1.04944.0100 | metal can         | 100g  |
| Oxidizing Digestion Agents Iodates/Periodates                  | Sodium chlorate pure   | EM1.06420.1000 | plastic bottle    | 1kg   |
|  | * Typically together with hydrochloric acid and/or nitric acid   |                |                   |       |
|  | Potassium iodate for analysis EMSURE® ACS,ISO,Reag. Ph Eur   | EM1.05051.0500 | plastic bottle    | 500g  |
|  | Sodium iodate for analysis EMSURE®   | EM1.06525.0100 | glass bottle      | 100g  |
|  | Sodium metaperiodate for analysis EMSURE® ACS,Reag. Ph Eur   | EM1.06597.0050 | plastic bottle    | 50g   |
| Oxidation with Chromium (VI) oxide, chromates and dichromates  | Potassium periodate for analysis EMSURE®   | EM1.05091.1000 | plastic bottle    | 1kg   |
|  | Potassium peroxodisulfate for analysis EMSURE®   | EM1.00229.0250 | glass bottle      | 250g  |
|  | Chromium(VI) oxide for analysis EMSURE®  | EM1.04952.0250 | plastic bottle    | 250g  |
|  | Potassium chromate for analysis EMSURE® ACS,Reag. Ph Eur   | EM1.04865.0500 | glass bottle      | 500g  |
| Oxidation with Hydrogen Peroxide H <sub>2</sub> O <sub>2</sub> | Potassium dichromate for analysis (max. 0.00001% HG) EMSURE® ACS,ISO   | EM1.04864.0500 | plastic bottle    | 500g  |
|  | Potassium dichromate for analysis EMSURE® ACS,ISO,Reag. Ph Eur   | EM1.06336.0250 | plastic bottle    | 250g  |
|  | Sodium dichromate dihydrate for analysis EMSURE® ACS   | EM1.07209.0250 | plastic bottle    | 250ml |
|  | Hydrogen peroxide 30% (Perhydrol®) for analysis EMSURE® ISO  | EM1.07210.0250 | plastic bottle    | 250ml |
| Oxidative Digestion with peroxide or peroxide sulfates         | Hydrogen peroxide 30% (Perhydrol®) (stabilized for higher storage temp.) for analysis EMSURE® ISO  |                |                   |       |
|  | * For the oxidation of organic and inorganic substances. Advantage: contamination free and matrix-free digestion. Oxidation both in acid and alkaline media. |                |                   |       |
|  | Sodium peroxide granular for analysis ACS,ISO  | EM1.06563.0100 | plastic bottle    | 100g  |
|  | Ammonium peroxodisulfate for analysis EMSURE® ACS,Reag. Ph Eur   | EM1.01201.0500 | plastic bottle    | 500g  |
|  | Potassium peroxodisulfate for analysis (<= 0.001% N) EMSURE® ACS,Reag. Ph Eur  | EM1.05092.0250 | plastic bottle    | 250g  |
| Fluxing with Hydrogen Sulfates/disulfates                      | Potassium peroxodisulfate for analysis EMSURE®   | EM1.05091.1000 | plastic bottle    | 1kg   |
|  | Sodium peroxodisulfate for analysis EMSURE®  | EM1.06609.0500 | plastic bottle    | 500g  |
|  | * Persulfates are used to convert Cr(III) to Cr(VI) and Mn(II) to permanganate   |                |                   |       |
|  | Potassium disulfate (Potassium pyrosulfate) for analysis EMSURE® ACS   | EM1.05107.1000 | plastic bottle    | 1kg   |
| Potassium hydrogen sulfate for analysis EMSURE® Reag. Ph Eur   | EM1.04885.0500   | plastic bottle | 500g              |       |
| Sodium hydrogen sulfate monohydrate for analysis EMSURE®       | EM1.06352.0500   | plastic bottle | 500g              |       |

## TIPS and Tricks

### Acid Digestion Agents

Acids are typically the first solvent to be used as they lead to minimal interference to subsequent analysis. If a residue remains after acid digestion, the sample may need to be treated with **alkali or special reagents** and further digested and analyzed.

### Alkaline Digestion Agents

Used when digestion proceeds rapidly at relatively **low temperatures (ca. 350–500 °C)**. Examples include Hydroxides (caustic alkalis and solutions), Alkaline metal carbonates and calcium carbonate.

### Oxidizing Digestion Agents

Normally used **in combination with alkaline metal hydroxides or carbonates**. Examples include nitrates, chlorates (in combination with HCL and/or HNO<sub>3</sub>), iodates, periodates, Chromium (VI) oxide, chromates and dichromates.

### Oxidizing Digestion with H<sub>2</sub>O<sub>2</sub>

For the oxidation of inorganic and organic substances. The advantage is a **contamination-free and matrix-free digestion**. Oxidation both in acid and alkaline media.

### Oxidizing Digestion with peroxide or peroxide sulfates

Persulfates are used to convert **Cr(III) to Cr(VI) and Mn (II) to permanganate**.



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