

Extracting Nitrosamines and 1-4 Dioxane: The Answers to Sample Prep Challenges for a Robust Analysis by GC

Nitrosamines and 1-4 dioxanes are compounds classes notorious both for their carcinogenic potential and for their widespread pollution throughout the environment. From air, to water, to food—they pose a concerning health hazard for humans. This has resulted in an increased interest in more efficient and reliable testing methods for robust analysis and determination of such compounds.

Lab analysts have reached out to Phenomenex technical experts to consult on challenges they experience when working with these types of compounds, from sample preparation to analysis. We've selected the most critical questions and provided the answers, which are summarized in this article in a Q&A format.



I am using a traditional SPE sample preparation and cannot get the level of extraction I need for nitrosamine or 1,4-Dioxane. What could you suggest?

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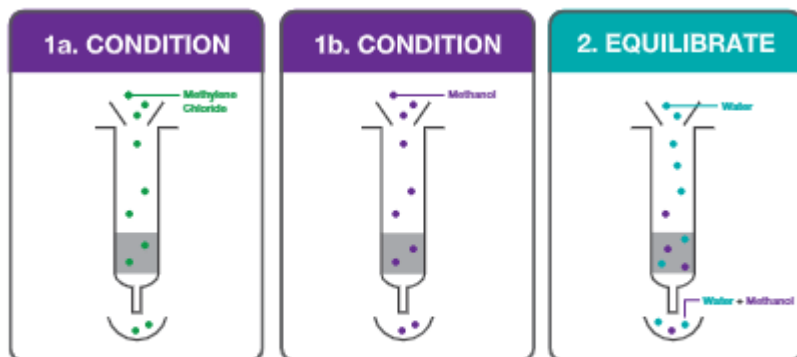
Due to the high polarity, when dealing with polar molecules like nitrosamines or 1-4 dioxane, traditional sample preparation using solid Phase Extraction (SPE) might not be efficient. Since the analyte is polar, a specialized extraction technique, such as activated carbon, is needed. Activated carbon has demonstrated the reproducibility and robustness in the extraction of polar analytes from aqueous matrices. Its high surface area combined with dense network of internal pore results in adsorption of analytes to its surface and eventually results in efficient extraction. Adsorption is a process whereby molecules stick to the surface area only. Activated carbon is large surface area and highly porous material. The analyte of interest easily gets adsorbed to the surface of the carbon particles by adsorption.

Are there any specific requirements for retention and elution of analytes such as nitrosamine 1,4-Dioxane on Strata Activated Carbon?

Regarding the elution, the sample volume and choice of solvent can be modified per analyte and method requirement. Generally, water-soluble polar analytes are good candidates for extraction on Strata Activated Carbon. A water insoluble solvent is recommended for elution. Nitrosamines are volatile, so adding a few drops of ethylene glycol to the extract will prevent evaporation and loss of analyte during the drying and reconstitution step. Recommended elution solvent is Methylene Chloride.

Here are the general extraction steps using Strata Activated Carbon:

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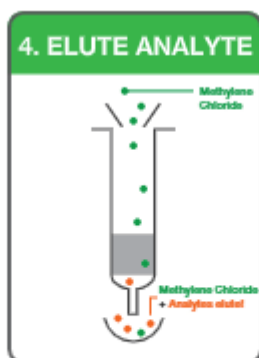
2 x 10 mL
Methylene Chloride

2 x 10 mL Methanol

2 x 10 mL Water



500 mL water sample
spiked with internal
standard. Dry for 10 min.



3 x 3 mL
Methylene Chloride

5. REMOVE MOISTURE

Pass the eluent through Methylene Chloride prewetted Strata Sodium Sulfate Giga™ Tubes, 5 g / 20 mL (Part No. [8B-S124-LEG](#)) and wash with 5 mL Methylene Chloride.

6. RECONSTITUTE

Evaporate solvent under Nitrogen to required volume and reconstitute with Methylene Chloride.

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1. CONDITION	2x 10 mL Methylene Chloride 2x 10 mL Methanol
2. EQUILIBRATE	2x 10 mL Water
3. LOAD	500 mL water sample spiked with internal standard Dry for 10 min
4. ELUTE	3x 3 mL Methylene Chloride
REMOVE MOISTURE	Pass the elute through Methylene Chloride prewetted Strata Sodium Sulfate Giga™ tubes, 5 g/20 mL (Part No. 8B-S124-LEG) and wash with 5 mL Methylene Chloride
RECONSTITUTE	Evaporate solvent under nitrogen to required volume and reconstitute with Methylene Chloride

Many laboratories do not want (or are not allowed) to use methylene chloride or other halogenated solvents to avoid environmental hazard. Is there an alternative eluting solvent for eluting analytes from Strata Activated Carbon?

Methylene chloride is used in the last elution step and is low volume. The general protocol for loading and elution on Strata Activated Carbon were based on EPA methods 521, and 522. Considering environmental perspective, MTBE and/or Ethyl acetate can be used as the extraction solvent.

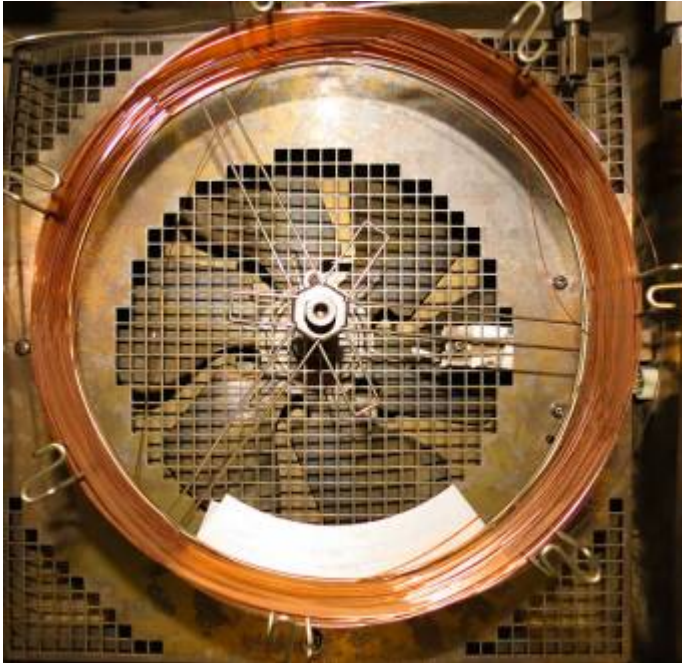
Can I use Strata Activated Carbon for the extraction of Nitrosamines from a Losartan drug sample? If so, can you provide additional information about sample

pre-treatment and which GC column you recommend for this analysis?

Yes, you can definitely use Strata Activated Carbon sample preparation as it has proven to be effective for Nitrosamine method (**TN-0154**). Regarding sample pre-treatment, Strata Activated Carbon adsorption-based cartridge and requires the max volume to slowly send the analyte and get max adsorption. 400mg cartridge 's max volume is 100ml water & the 2g is 500ml. Hence, for the 400mg Activated carbon, Losartan API can be dissolved in 100mL water to facilitate adsorption. If the formulation is water insoluble, small volume of Organic can be added (1-2mL) followed by 100ml water dilution and spun down undissolved material, then pass the centrifugate through the cartridge. The analyte of interest- Nitrosamine will still be soluble in aqueous extract & will go through the cartridge even though there were undissolved components from formulation.

Regarding a GC column for this specific analysis, the Zebron ZB-5MSplus is designed with a rigorous fused silica deactivation process that improves inertness for troublesome compounds to reduce potential surface activity. We used the Zebron ZB-5MSPlus in a similar study. The column delivers robust analysis due to its specialized deactivation for versatile 5% phenyl-arylene selectivity with improved sensitivity, and low bleed (MS Certified).

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I am using Strata Activated Carbon for a retaining 1,4-Dioxane and I am looking for a recommendation on a GC column than could provide the best resolution. What can you recommend?

The GC column Zebtron ZB-624PLUS is the optimal choice for the analysis of 1,4- Dioxane. The superior deactivation process enhances peak shape, improves signal-to-noise levels, and increases sensitivity for qualitative and quantitative analysis of active compounds. This column is the “go to” choice for volatile compound analysis in environmental samples.

Feel free to reach out to our Technical Experts with questions and assistance on method development for this and other studies. [Chat Now.](#)

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More resources:

Strata Activated Carbon sample prep, Zebron columns ZB-624plus and ZB-5MSplus have demonstrated high resolution and robustness when resolving polar compounds. Data on product performance comparison is available in the technical notes listed below. Click to view and download:

- [Strata Activated Carbon 2g/6mL: Extraction and Robust Analysis of 1,4-Dioxane by Isotopic Solution](#)
- [Strata Activated Carbon 2g/6ml: Extraction and Robust Analysis of Nitrosamines from Water Sample](#)
- [Strata Activated Carbon 400 mg pass-through: Extraction and Robust Analysis of Acrylamide in Coffee](#)
- [Strata Activated Carbon Product Brochure](#)

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