Conveniently Evaluate GC Inlet Inertness with Endrin & DDT Using Our Ready-to-Use Reference Standard

There are a variety of ways to determine the inertness of a chromatographic system. One of the more stringent tests for GC inlets uses low concentrations of dichlorodiphenyltrichloroethane (DDT) and endrin as inertness probes. These pesticides appear in many environmental methods as analytes of interest, but for analysts using instruments sensitive enough to detect them in the ppb and ppt range (e.g., GC-ECD, GC-MS/MS), they can also serve as sensitive probes of GC inlet inertness. For less sensitive detectors (e.g., GC-FID), where detectable concentrations of endrin and DDT are much higher, they are less useful. At concentrations in the low parts per billion range, however, these two compounds are excellent at revealing just how inert a GC inlet really is. Restek offers a convenient reference standard that is prepared at the appropriate concentrations and is ready to use without further dilution.

Why Endrin & DDT?

Endrin and DDT make excellent indicators of inlet inertness. If the sample pathway in the GC isn't exceptionally inert, endrin and DDT are very likely to undergo chemical reactions within a hot GC inlet. Depending on the type and degree of active site present in a GC inlet, endrin and DDT response will decrease as those molecules are converted into different compounds through chemical reaction. The evidence of this change will typically appear as distinct peaks in the chromatogram.

The chemical reactions produce predictable products. For endrin, endrin aldehyde and endrin ketone result from the opening of the epoxide ring found within endrin, especially in the presence of acidic active sites. DDT can undergo conversion to DDE (dehydrochlorination) and DDD (dechlorination). In cases of extreme inlet activity, endrin and DDT can be completely converted into these and other products that represent further reactions occurring in the inlet.

In an inlet with active sites capable of reacting with endrin or DDT, the products of these chemical reactions are formed prior to the analytical GC column, which is why they can appear as distinct and frequently symmetrical peaks. These unwanted products are created before reaching the analytical column in the inlet. When they then reach the analytical column, they are subjected to chromatographic separation along with every other compound injected into the instrument.

The good news is because these products appear as distinct peaks, they can be monitored. Their presence can be used to evaluate the degree of breakdown that occurred as a result of GC inlet activity, and this measurement is a strong marker of the overall inertness of a GC inlet.

One example of a method that uses this behavior is US EPA Method 508.1. This method, along with other similar methods, has a protocol for qualifying an instrument before use. This protocol ensures an instrument is inert enough to analyze samples for the presence of pesticides. Significantly, this technique can be helpful for more analysts than those running this specific method.

Using 508.1 Degradation Mix to Assure an Inert Analytical Pathway

US EPA Method 508.1 is a disk liquid-solid extraction, flash injection capillary column gas chromatography (GC) technique using an electron capture detector (ECD). This makes it extremely sensitive to halogenated compounds like endrin and DDT. Low concentrations (pg on-column) of halogenated pesticides in drinking (finished) water can be determined using this method. The pesticide degradation (breakdown) check is performed prior to analysis and at least every 12 hours during a sequence of samples. This check involves injecting a standard of endrin and DDT and calculating what is known as the "breakdown percentage." Restek offers a reference standard, the 508.1 Degradation Check Mix, which can be used to test for inlet inertness. This standard can be used without any further dilution since it is already at the desired concentration levels for endrin and DDT.

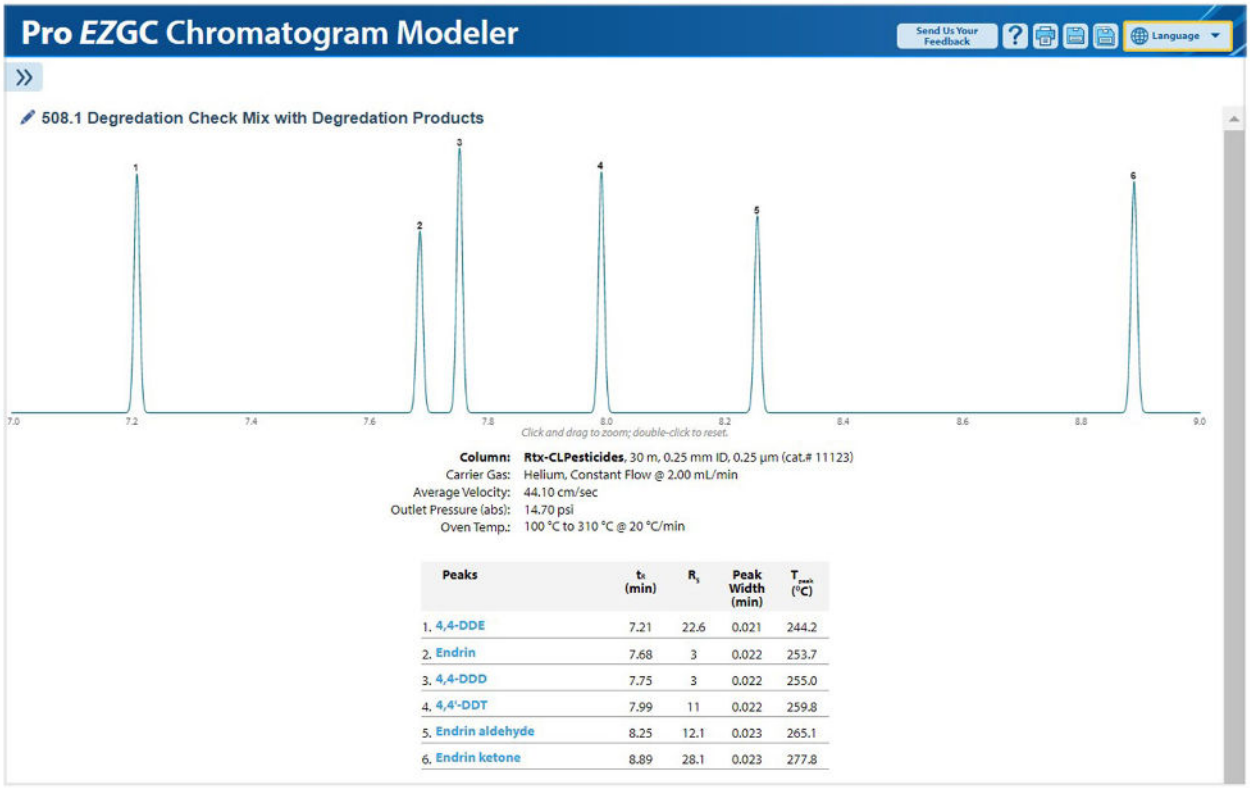
Breakdown is calculated as follows:

$$\text{Endrin Breakdown \%} = \left(\frac{\text{Endrin Aldehyde Area} + \text{Endrin Ketone Area}}{\text{Endrin Aldehyde Area} + \text{Endrin Ketone Area} + \text{Endrin Area}} \right) \times 100\ \%$$

$$\text{DDT Breakdown \%} = \left(\frac{\text{DDD Area} + \text{DDE Area}}{\text{DDD Area} + \text{DDE Area} + \text{DDT Area}} \right) \times 100\%$$

In the case of US EPA 508.1, if degradation of either DDT or endrin exceeds 20%, inlet and column maintenance is required to restore performance. This is a good rule of thumb for anyone interested in using this reference standard to evaluate a GC inlet's inertness, whether you're performing US EPA 508.1 or not. For your specific method and Restek column, Restek's Pro *EZ*GC chromatogram modeler may be helpful to determine the elution times for these components. Endrin and DDT must be resolved from their breakdown components to assure accurate calculations (see figure 1).

Figure 1: Restek's Pro *EZ*GC software displaying elution times.



Conclusion

Endrin and DDT were chosen specifically for US EPA 508.1 because they are sensitive to active sites within a hot injector, and they require highly inert conditions to pass through the inlet without breaking down. The performance of these pesticides has implications beyond DDT and endrin inertness. Poor performance (high breakdown) for these pesticides is an indication that there will be similar issues with other sensitive analytes. Laboratories that do not perform environmental work can also use this test as a verification of an inert sample pathway. Inert liner deactivations, such as Restek's Topaz liners, use complex chemistries to prevent analyte interaction and breakdown in the inlet. Every lot of Topaz liners are tested using DDT and endrin to demonstrate they are ready for the most demanding applications. Restek's 508.1 Degradation Check Mix is a convenient tool for evaluating the inertness of your GC's inlet.

If you have any questions about how to use endrin and DDT as inlet probes, or if you need help with your sample preparation or chromatography, contact your local Restek Sales representative.

508.1 GC Degradation Check Mix

(2 components)

4,4'-DDT (50-29-3) Endrin (72-20-8)

Conc. in Solvent	CRM?	Min Shelf Life on Ship Date	Max Shelf Life on Ship Date	Shipping Conditions	Storage Temp.	qty.	cat.#
100 µg/mL each in ethyl acetate, 1 mL/ampul	Yes	6 months	52 months	Ambient	10 °C or colder	ea.	32093

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Questions? Contact us or your local Restek representative (www.restek.com/contact-us).

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