National Institute of Standards & Technology

Report of Investigation

Reference Material 8469

4,4'-DDT

This Reference Material (RM) is provided as a primary reference compound of measured purity for 4,4'-DDT. It is intended for use in the evaluation of procedures used in the measurement of 4,4'-DDT in environmental samples and for the preparation and evaluation of daily working standards used in these procedures. This material can also be used in any case when 4,4'-DDT of known purity is required. A unit of RM 8469 consists of one vial containing approximately 100 mg of 4,4'-DDT.

IDENTIFICATION AND PURITY INVESTIGATION

The purity of 4,4'-DDT and the estimated uncertainty are given in Table 1. The purity listed is based on analyses by gas chromatography (GC), differential scanning calorimetry (DSC) and the Karl Fischer method for the determination of water. Identification is based on mass spectral analysis and melting point determination. GC retention times on two capillary columns with different polarity were used as further confirmation. It is important to note that this is **not** a Standard Reference Material (SRM) and the purity value presented in Table 1 is not certified.

Table 1. Percent Purity of 4,4'-DDT

Chemical Abstracts Services (CAS) Nomenclature	CAS Registry Number	Purity (weight percent)
1,1'-(2,2,2-trichloroethylidene)bis[4-chlorobenzene]	50-29-3	99.8 ± 0.1

The purity and the estimated uncertainty interval are based on scientific judgment and evaluation of the analytical procedures applied on this material including the analytical imprecision and the systematic differences between the two methods (GC and DSC).

NOTICE AND WARNING TO USERS

Handling: This compound has been reported to have toxic, mutagenic and/or carcinogenic properties when ingested, inhaled or absored into the skin. Handle with care, use proper protective gear, use proper disposal methods and wash hands after handling.

Storage: Vials, as received, should be kept tightly sealed, protected from the light, and stored in a refrigerator or freezer.

Preparation and analytical determinations were performed in the NIST Organic Analytical Research Division by R. E. Rebbert, N.E. Gallauresi, S.A. Margolis and R.M. Parris.

The coordination of the technical measurements for determination of the purity was under the direction of R.E. Rebbert, R.M. Parris and S.A. Wise of the NIST Organic Analytical Research Division.

Gaithersburg, MD 20899
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William P. Reed, Chief
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Consultation on the statistical design of the experimental work and on evaluation of the data was provided by S.B. Schiller, Statistical Engineering Division.

The technical and support aspects involved in the preparation and issuance of this Reference Material were coordinated through the Standard Reference Materials Program by T.E. Gills.

PREPARATION AND ANALYSIS

4,4'-DDT, used in the preparation of this RM, was obtained from Crescent Chemical Company, Hauppauge, New York. The bulk material was dispensed into 4 mL amber glass vials. Ten vials were then chosen for analysis using a modified stratified random selection. Two samples of about 1 mg each were taken from each vial for the DSC measurements of purity and two additional samples of about 3 mg each were taken from each vial for the GC measurements of purity using a flame ionization detector (FID). These latter samples were dissolved in hexane and then 2 to 3 μL were injected for the GC analyses. GC analyses were performed on two columns with different polarity, i.e., a 5 % phenyl-substituted methylpolysiloxane (DB-5) and a 14 % cyanopropyl phenyl-substituted methylpolysiloxane (DB-1701). A summary of the results of the measurements for each method is given in Table 2.

Both of these methods (GC and DSC) have limitations. In the GC method with FID, assumption is made that the parent compound and the impurities have equal area response factors on a weight basis. Errors can result from 1) the retention of some impurities on the column or 2) insufficient resolution between the impurities and 4,4'-DDT. Both of these effects would tend to give purity results that are too high. Two GC columns with different polarity were used to try to minimize these two effects. A third source of error is the partial decomposition of the parent compound during analysis. This effect would tend to give purity results that are too low. There is evidence in the literature that 4,4'-DDT decomposes under the GC conditions used, but this effect is minimal since the purity values determined by GC are already slightly greater than that determined by DSC. Moreover, the product of the thermal decomposition is 4,4'-DDE and this is found at a maximum of 0.04 % as an impurity. The DSC method can also introduce systematic errors caused by the formation of mixed crystals. This would result in a purity value that is higher than the actual value. The DSC method produces purity results on a molar basis. However, the result is approximately equal to a weight basis if the impurities have the same molecular weight as the main constituent.

Water analysis was performed on three samples of about 100 mg each using the Karl Fischer method. Within the experimental error (0.05 weight %), no water was found in this RM and therefore, no correction was made for moisture content.

SUMMARY

The value of purity presented in this report, as listed in Table 1, is based on measurements using gas chromatography, differential scanning calorimetry and Karl Fischer water analysis. It is the mean of the results of the three (3) employed methods. The purity is a "best estimate" value since in addition to the approximations mentioned above, no analyses were made for inorganic or insoluble substances.

Table 2. Summary of Analytical Results

Analytical Method	Purity
DSC	99.73 ± 0.03 (mole percent)
GC (DB-5)	99.84 ± 0.01 (weight percent)
GC (DB-1701)	99.81 ± 0.02 (weight percent)

Each uncertainty interval listed is the standard deviation of a single measurement based on 20 measurements for each method.