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**Pacific Northwest  
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# Evidence That Certain Waste Tank Headspace Vapor Samples Were Contaminated by Semivolatile Polymer Additives

J. L. Huckaby

February 2006



Prepared for the U.S. Department of Energy  
under Contract DE-AC05-76RL01830

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Richland, WA 99352

## **Summary**

Vapor samples collected from the headspaces of the Hanford Site high-level radioactive waste tanks in 1994 and 1995 using the Vapor Sampling System (VSS) were reported to contain trace levels of phthalates, antioxidants, and certain other industrial chemicals that did not have a logical origin in the waste. This report examines the evidence these chemicals were sampling artifacts (contamination) and identifies the chemicals reported as headspace constituents that may instead have been contaminants. Specific recommendations are given regarding the marking of certain chemicals as suspect on the basis they were sampling manifold contaminants.

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## 1.0 Introduction

Vapor samples collected from the headspaces of the Hanford Site high-level radioactive waste tanks in 1994 and 1995 using the Vapor Sampling System (VSS) were reported to contain trace levels of phthalates, antioxidants, and certain other industrial chemicals that did not have a logical origin in the waste. Phthalates are commonly used as plasticizers<sup>1</sup> and their presence in the samples was considered evidence some plastic component of the sampling system was outgassing phthalates into the air stream being sampled. However, they were not considered an important finding at the time, and their origin was not investigated. Recent efforts to identify headspace chemicals that could potentially be hazardous to tank farm workers have indicated two of the phthalates and several other related chemicals to be of potential concern. This report examines the evidence these chemicals were sampling artifacts (contamination) and identifies the chemicals reported as headspace constituents that may instead have been contaminants.

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<sup>1</sup> Plasticizers are chemicals added to the formulation of plastics to improve their flexibility, machinability, finish, or other property. To reduce the evaporative loss of plasticizers from a product, they are typically semivolatile compounds.

## 2.0 Background

The systematic characterization of tank headspace gases and vapors began in 1994 with the deployment of the VSS. It was a system to collect vapor samples using air transfer tubing, a valved manifold, air flow meters, and an air pump. The VSS pulled air from the headspace of a tank and exposed sampling devices (i.e., sorbent traps and SUMMA canisters) to the air stream in a controlled fashion. To prevent condensation of water vapor and reduce the adsorption of vapors on the manifold's inner surfaces, all components of the manifold upstream of the sampling devices were heated about 20 °C above the tank headspace temperature or typically 50 °C (e.g., Caprio 1995).

To demonstrate the VSS was free of contamination from volatile cleaning solvents and residual vapors from previous sampling events, a SUMMA canister sample of ambient air was collected using the VSS before it was connected to the tank headspace. This consistently demonstrated only low levels of few, if any, volatile organic vapors to be present in the sampling system. This was not, however, a meaningful indicator of the presence or lack of semivolatile vapors, because the SUMMA analysis conducted at the time was not suitable for low levels of semivolatile vapors.

Procedures required that after it had been connected to the tank headspace and the desired system temperature had been reached the VSS was purged with air from the tank headspace for about 30 minutes at a flow rate of just over 5 L/min. This was intended to replace the air in the system with air from the tank headspace, bring the adsorbed species on the inner surfaces of the system into equilibrium with the headspace air, and further reduce any residual volatile vapor contamination in the system.

To prevent radiological contamination of the samples being collected and the VSS itself by airborne radioactive particles<sup>2</sup>, air from the headspace was drawn through two to four 47-mm glass fiber filters. The filters were removed and assayed after each sampling job or as needed for the shipment of samples, and new filters installed for subsequent sampling. Because the glass fiber filters were known to have a relatively high surface area and were considered a likely adsorption sink for semivolatile organic vapors present in the headspace air stream, the filter holders were maintained at much higher temperatures than the rest of the VSS – the sampling report for tank C-103 indicates 100 to 150 °C was the desired temperature (Mahon et al. 1994).

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<sup>2</sup> No radioactive gases or vapors of consequence were expected. Radioactive particles were limited to radon daughters that decayed to negligible levels in about one day.

### 3.0 Contamination from the Sampling Manifold

Use of the glass fiber filters was identified as a potential source of contamination in December 1995<sup>3</sup>. Though interest at that time appears to have been in volatile (organic solvent) contamination, the potential for volatiles on the filter implies the filters being used were a type that contained an acrylic binder. This is important because the acrylic binder may have contained plasticizers and/or antioxidants that would have been released during the sampling event while the filter was at an elevated temperature (100 to 150 °C) and collected in the samples. Subsequent vapor sampling events using the VSS and its replacement, the In Situ Vapor Sampling (ISVS) system, were conducted with filters that had been “baked out” to minimize contamination<sup>4</sup>.

In addition to the potential contamination from the filter itself, the glass fiber filters were sealed within their filter housings with an o-ring (typically Viton<sup>®</sup> but possibly some variety of Buna) and vacuum grease applied to the o-ring. The stainless steel filter housing also contains a hard plastic retainer ring, though this could only contaminate the manifold if the filter was not sealed properly. The current version of the retainer ring sold by the Pall Corp. is polyfluorotetraethylene (PFTE) but other materials, such as Nylon, may have been used in 1994 and 1995<sup>5</sup>. The outgassing of plasticizers by the o-ring and the retainer ring at the elevated temperature of the filter housing is a potential additional source of the plasticizers detected in the samples.

Plasticizer contamination may have also come from the plastic ferrules used to seal miniature thermocouple wells in the VSS. These were 0.125-in. perfluoroalkoxy (PFA) Teflon<sup>®</sup> or Nylon Swagelok<sup>®</sup> ferrules that would have been exposed to the air sample stream at the temperature of the manifold (40 – 50 °C).

Evidence that plasticizers and other chemicals associated with the fabrication and preservation of the filter, o-ring, retainer ring, and thermocouple seals is considered here to be of four types.

1. The chemicals in question are neither known waste constituents nor reasonable waste chemistry products.
2. Analyses of similar materials (e.g. Viton o-rings) at elevated temperatures has indicated the presence of some of the chemicals in question.
3. The chemicals in question were reported primarily in samples collected with the VSS (where elevated temperatures were used), and observations of plasticizers are correlated with each other (several plasticizers are frequently seen together) and/or in time (certain plasticizers are seen in each tank that is sampled during a given period of time).
4. Subsequent vapor samples from a tank identified as having plasticizers in its headspace have failed to corroborate the original finding. Also, of the chemicals in question, only the phthalates have been detected in condensed-phase waste samples.

Each type of evidence is discussed below.

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<sup>3</sup> Email from Ricky D. Mahon to Thomas C. Tribble, David A. Dodd, Larry L. Lockrem, James L. Huckaby, and Luther L. Buckley on 12/21/1995 entitled “Problem getting baked filters” indicates the Gelman Scientific, Inc., filters being used were found by an unnamed Gelman client to introduce chemical contamination.

<sup>4</sup> Email from Ricky D. Mahon to Thomas C. Tribble, Luther L. Buckley, David A. Dodd, James L. Huckaby, and Larry L. Lockrem on 1/2/1996 states that the glass fiber filters were being baked out for use in the January 1996 vapor sampling events, and that pre-baked filters had been ordered from the filter supplier, Gelman Scientific, Inc.

<sup>5</sup> Gelman Scientific, Inc. instrumentation is now sold by the Pall Corporation, East Hills, New York.



### 3.1 Distinctive Properties of Sampling Manifold Contaminants

Stock (2004) examined the origins of organic compounds in the waste tanks, including those species known to have been put into the tanks and those species that are being formed by chemical reactions within the waste. Nearly all of the organic vapors detected in the tank headspaces can be explained in terms of these two origins, with the exceptions primarily being the semivolatile antioxidants and plasticizers discussed in this letter.<sup>6</sup> Stock reviewed the waste chemistry and has proposed mechanisms for the formation of the most common organic waste degradation products, e.g., alkanes, alkenes, alcohols, ethers, aldehydes, ketones, acids, esters, nitriles, nitrates, nitrites, nitro- compounds, etc. A common organic degradation reaction scheme involves the formation of a radical (usually via ionizing radiation) followed by oxidation of the fragments. These classes of compounds are well represented, with most and sometimes all members of the homologous series of straight C1 through C10 chain molecules having been reported in samples from the tank headspaces.

Chief among the species whose origins are not explainable, either as chemicals known to have been sent to the waste tanks or as products of the waste chemistry, are various molecules used as antioxidants, several phthalates, and several sulfonamides. These are listed in Table 1, along with their Chemical Abstracts Services (CAS) registry numbers, molecular weights, maximum reported headspace concentrations, the number of tanks in which they have been reported, and notes on their industrial plasticizer uses.

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<sup>6</sup> Other chemicals found in the headspaces that currently lack satisfactory explanations include several volatile halocarbon compounds.

**Table 3.1.** Semivolatile Chemicals without Identified Waste Origins

	CAS <sup>(1)</sup> Number	MW <sup>(2)</sup> (g/mol)	Max. Conc. (ppmv)	Number of Tanks	Common Names – Notes
<b>Phenols</b>					
4-tert-Butyl phenol	98-54-4	150.22	0.0013	3	Rylex 30 – Vulcanizing agent and/or antioxidant in rubber, auxiliary cross-linking agent for halobutyl rubber.
2-tert-Butyl-4-methoxyphenol	121-00-6	180.25	0.00050	1	Butylated hydroxyanisole, BHA – One of the two BHA isomers; antioxidant and stabilizer commonly used in cosmetics, food packaging, and foods such as vitamins, oils, waxes, sausage, chewing gum, shortening, and lard.
tert-Butyl-4-methoxyphenol	25013-16-5	180.25	0.00037	1	Butylated hydroxyanisole – Isomeric mixture of 2-tert-butyl-4-methoxyphenol and 3-tert-butyl-4-methoxyphenol. See comments for 121-00-6.
2,6-Di-tert-butyl-4-methylphenol	128-37-0	220.36	0.51	3	Butylated hydroxytoluene, BHT – Antioxidant for plastics, elastomers, petroleum, food, chewing gum, etc.
Di-tert-butyl-ethylphenol	4130-42-1 <sup>(3)</sup>	234.38	0.00041	1	Butylated hydroxyethylbenzene, BHEB – Antioxidant for natural and synthetic rubbers, acrylonitrile butadiene rubber, polychloropropene rubber, and latex. Stabilizer for polyolefins.
<b>Phthalates</b>					
Diethyl phthalate	84-66-2	222.24	0.13	13	DEP – Plasticizer, fragrance fixative, nail polish plasticizer, used in deodorants; very common plasticizer.
Dibutyl phthalate	84-74-2	278.35	0.00048	2	DBP – Plasticizer, softener for natural rubber, synthetic rubber (particularly polychloropropene), acrylonitrile butadiene rubber, polyvinyl acetate, etc.
Butyl cyclohexyl phthalate	84-64-0	304.39	0.0059	1	Processing aid for molded ethyl cellulose products, PVC plasticizer, etc.; plasticizer for polymers, elastomers, and nitrocellulose lacquers.
Butyl 2-methylpropyl phthalate	17851-53-5	278.35	0.00085	1	No information found for this specific chemical. Possible contaminant or coproduct of dibutylphthalate.
Butyl 2-ethylhexyl phthalate	85-69-8	334.46	0.00060	1	BOP – Primary phthalate plasticizer for vinyl chloride resins and copolymers; used in wire and cable insulation, etc.
bis(2-Ethylhexyl) phthalate	117-81-7	390.57	0.00029	1	DEHP, DOP – One of the most widely used of all vinyl plasticizers.
<b>Other Benzene Derivatives</b>					
Methyl 4-tert-butylbenzoate	26537-19-9	192.26	0.00052	1	MBB – A melamine hardener for resin powder coatings, and an intermediate for 4-tert-butyl-p-methoxydibenzoylmethane (UVA filter).
N-Phenylbenzenamine	122-39-4	169.23	0.030	10	Used in the laboratory as a Redox indicator. Used as stabilizer and antioxidant in natural and synthetic rubbers; extends life of transmission fluids, lubricants, hydraulic fluids exposed to high-temperature or high-loads.
Phenyl-a,a-dimethylmethanol	617-94-7	136.20	2.2	5	Reported by Manura (1994) in Viton o-ring off-gases.

**Table 3.1.** Semivolatile Chemicals without Identified Waste Origins

	CAS <sup>(1)</sup> Number	MW <sup>(2)</sup> (g/mol)	Max. Conc. (ppmv)	Number of Tanks	Common Names – Notes
<b>Sulfonamides</b>					
N-Butylbenzenesulfonamide	3622-84-2	213.30	0.16	39	BSA – Used in Nylon 6, Nylon 11, Nylon 12, cellulose acetate, molded polyamide parts. Improves Nylon removal from molds, machining properties, and finish.
N-Ethyl-4-methylbenzenesulfonamide	80-39-7	199.27	0.0014	1	Plasticizer for polyamides, shellac, cellulose acetate, etc.
N-Hexylbenzenesulfonamide	7250-80-8	241.35	0.00043	1	No information specific to this compound. Probable component of proprietary plasticizer formulation.
C6-Benzenesulfonamide	n.a.	241.35	0.00035	1	No information specific to this compound. Probable component of proprietary plasticizer formulation.
<b>Others</b>					
1-(1,1-Dimethylethyl)-2-methyl-1,3-propanediyl 2-methylpropanoate	74381-40-1	286.42	0.45	12	No information on this specific compound found. It is nearly identical to TXIB (CAS 6846-50-0) a proprietary (Eastman Chemical) plasticizer; it may be misidentified by mass spectral library fit or may be used to circumvent Eastman Chemical patents on TXIB.
2,6-bis(1,1-Dimethylethyl)-2,5-cyclohexadiene-1,4-dione	719-22-2	220.31	0.0029	8	2,6-di-tert-butyl-p-benzoquinone, DTBB – Antioxidant, melting point 65 – 67 °C.

1. CAS = Chemical Abstracts Service.
2. MW = molecular weight.
3. The CAS number given is that of the antioxidant 2,6-Di-tert-butyl-4-ethylphenol.

## 3.2 Laboratory Analyses of Sampling Manifold Components

No chemical analyses were conducted at Hanford in the period of interest to address the outgassing of semivolatiles from the plastic and rubber VSS components. However, studies have been conducted to examine the contamination of food and pharmaceutical products by contact with similar materials, and the outgassing of o-ring seals and plastic components are issues for ultra-clean and high vacuum systems (Manura 1994, Wahl et al. 1996, Manura et al. 1997). In a study particularly relevant to this study, Manura (1994) examined the volatile and semivolatile components of Viton® and Buna-N o-rings by heating the o-rings to 150 °C. The major components he found are reported in Table 2. Those compounds that have also been reported in tank headspace samples in Table 2 are noted. Though the 150 °C test temperature used by Manura is probably at the high end of the VSS o-ring temperature range, the results are qualitatively applicable. Most of the chemicals identified in the Manura study for these two types of o-rings were reported in VSS samples, suggesting that the proposed explanation for their presence in the samples – that they were outgassed by the o-rings in the filter housings – is correct. Because the Manura study was probably not conducted on o-rings from the same manufacturer as the o-rings used in the VSS, the agreement is not perfect. Formulations of plasticizers and mold release agents used vary with manufacturer, and may vary from batch to batch.<sup>7</sup>

**Table 3.2.** Outgas Components from Viton and Buna-N O-Rings Heated to 150 °C (Manura 1994)

CAS <sup>1</sup> Number	Chemical	Note	O-Ring Material
3173-53-3	Isocyanatocyclohexane		Viton
95-50-1	1,2-Dichlorobenzene	2	Viton
104-76-7	2-Ethyl-1-hexanol	3	Viton
98-86-2	1-Phenylethanone	2	Viton
617-94-7	Dimethylbenzenemethanol	2	Viton
124-19-6	Nonanal	3	Viton
629-59-4	n-Tetradecane	3	Buna-N
107-50-6	Tetradecamethylcycloheptasiloxane		Buna-N
147-47-7	1,2-Dihydro-2,2,4-trimethylquinoline		Buna-N
25013-16-5	Butylated hydroxyanisole (BHA)	2	Buna-N & Viton
719-22-2	2,6-Di-tert-butyl-p-benzoquinone	2	Viton
629-62-9	n-Pentadecane	3	Buna-N
128-37-0	Butylated hydroxytoluene (BHT)	2	Buna-N & Viton
4130-42-1	2,6-Di-tert-butyl-4-ethylphenol		Buna-N
544-76-3	n-Hexadecane	3	Buna-N
84-66-2	Diethylphthalate (DEP)	2	Viton
122-39-4	N-Phenylbenzeneamine	2	Buna-N
629-78-7	n-Heptadecane	3	Buna-N
3910-35-8	2,3-Dihydro-1,1,3-trimethyl-3-phenyl-1H-indene		Buna-N & Viton
593-45-3	n-Octadecane	3	Buna-N
629-92-5	n-Nonadecane	3	Buna-N

1. CAS = Chemical Abstracts Services.
2. Reported in headspace samples and considered a potential sampling contaminant.
3. Reported in headspace samples but contamination levels not apt to significantly affect headspace concentrations.

<sup>7</sup> In a telephone conversation with a representative of duPont on October 10, 2005 it was learned that Viton brand o-rings are made by individual manufacturers from raw Viton supplied by duPont, and that formulations and mold-release agents vary depending on the manufacturer.

### 3.3 Plasticizer Observations

Table 3 lists all the vapor sampling events in chronological order and indicates, with black table cells, which of the Table 1 plasticizers were reported during each sampling event. The date and tank being sampled are given, as well as the sampling method used to collect samples. Gray shading has been used to indicate when the VSS was used to collect samples, a consideration here because the plastic components in the VSS were heated and plasticizer offgassing correspondingly greater. Table 2 indicates the following:

1. The plasticizers in question tend to have been reported in samples collected before January 1996 and in samples collected using the heated-manifold of the VSS. While it is true that these early analyses frequently provided lower detection limits, particularly for tentatively identified compounds, over 20% of the plasticizers reported before January 1996 were at concentrations above the highest subsequent reporting limits for tentatively identified compounds (10 ppb).
2. Several of the plasticizers appear in nearly every tank sampled during a period of time. N-butylbenzenesulfonamide, for example, was reported in 37 of 39 successive sampling events (from all 12 single-shell tank farms), but in only 2 other of the remaining events. This particular example also happens to have only been reported by one of the analytical laboratories, though the average level reported (about 14 ppb) would have been well above the reporting limits of other laboratories. Given 95% of the tanks found to have this chemical were sampled in a 1-year time period, it is more reasonable that the chemical was from some contamination source and not from the tank headspaces. Though the statistics are less dramatic for other plasticizers, the same observation generally applies – they tend to be reported in short streaks as might be expected if the sampling manifold or sampling methodology were subject to systematic contamination from new plastic manifold parts or unintended exposures during sample handling.
3. The plasticizers tend to be seen with other plasticizers. Ten plasticizers were reported in sampling event 36 at tank C-108, six were reported in event 41 at tank C-112, etc. This is in part due to the fact that lower detection limits were applied in some of the tanks, but the point remains that they tend to occur together. This suggests these chemicals were combined and used as plasticizers, and are not, for example, from an unrecorded process use of an antioxidant.

### 3.4 Consistency of Sampling Data

The tanks that were reported to have the plasticizers in the mid-1990s were not found to have the plasticizers when they were resampled. For example, none of the plasticizers reported in samples from tanks BY-108, C-107, and S-102 between September 1994 and March 1995 (1 event at each tank) have been found again in those tanks despite at least five subsequent sampling events at each tank. Though not all tanks reported to have plasticizers in their headspaces have been resampled, there has been only one incidence of a plasticizer reported in a tank more than once.<sup>8</sup>

Also, volatile and semivolatile organic analyses of condensed-phase waste samples have failed to identify any of the non-phthalate plasticizers. Analyses of liquid and solid waste samples have indicated the presence of diethylphthalate, dibutylphthalate, and bis(2-ethylhexyl)-phthalate (TWINS 2006).

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<sup>8</sup> N-Butylbenzenesulfonamide was reported in the August 1994 and March 1995 samples from U-106. This particular plasticizer, used in Nylon plastics, was also present in samples from 18 of the 22 tanks sampled between the two U-106 sampling events.

## 4.0 Recommendations

The presence of the plasticizers in vapor samples was not considered a safety issue in the 1990s and not addressed at the time the analytical results were received. Testing that could prove the source of the plasticizers was within the sampling manifold was not done at that time and can not be done now, because the sampling manifolds used during the 1990s have been dismantled and disposed of. Given the evidence described above, it is judged that the chemicals listed in Table 1 were from off-gassing of components in the sampling manifolds, and not from the tank headspaces. However, those phthalates that have also been reported in condensed-phase waste samples (diethylphthalate, dibutylphthalate, and bis-(2-ethylhexyl) phthalate) should be considered potential headspace constituents. It is therefore recommended that results associated with the “Phenols”, “Other Benzene Derivatives”, “Sulfonamides”, and “Others” in Table 1 be considered suspect on the grounds there are strong bases for them being contaminants of the sampling manifold.

## 5.0 References

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