A REPORT ON THE RMA TCLP ASSESSMENT PROJECT

PREPARED FOR
THE RUBBER MANUFACTURERS ASSOCIATION

WASHINGTON, D.C.

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20th★★★★ Anniversary

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1.0 INTRODUCTION

1.1 EXECUTIVE SUMMARY

PROJECT SCOPE: The Rubber Manufacturers Association (RMA) authorized Radian Corporation to assess what levels of chemicals, if any, are leached from representative RMA products using EPA's proposed Toxicity Characterization Leaching Procedure (TCLP). The TCLP proposes to add chemicals to the existing list of compounds regulated under Subtitle C of the Resource Conservation and Recovery Act, and to introduce new extraction methods.

RESULTS OF THE TCLP STUDY: None of the products tested, cured or uncured, exceeded proposed TCLP regulatory levels. Most compounds detected were found at trace levels (near method detection limits) from ten to one hundred times less than proposed TCLP regulatory limits. In only one test was a proposed limit approached. Methyl ethyl ketone (MEK), was detected in the TCLP leachate of a printer roller product at 7.0 mg/L. The proposed TCLP limit is 7.2 mg/L.

EP TOXICITY COMPARISON: Additional work was performed to compare the results of EP Toxicity procedures with the proposed TCLP for RMA products, the results from the two leachate methods were comparable.

EFFECTS OF LEACHING UNGROUND SAMPLES: Radian also compared the effect of a modification to the TCLP recently proposed by EPA which would eliminate grinding prior to leaching; in effect making TCLP tests of rubber products more representative of disposal practices. The results inherent in ground and unground samples were comparable. Uncertainties in the TCLP procedure had a greater impact on the variability of the results than differences in ground and unground methods.

1.2 SCOPE OF THE RMA STUDY

The Toxic Characteristic Leaching Procedure (TCLP) was proposed as an amendment to EPA's hazardous waste identification regulations (40CFR Parts 261, 271, and 302) on June 13, 1986 in the Federal Register/Vol. 51, No. 114. It is the intent of EPA to replace the current waste characterization method (EP Toxicity) with the TCLP.

1.2.1 THE TCLP STUDY

In anticipation of a change in waste characterization methodologies, the Rubber Manufacturers Association (RMA) authorized Radian Corporation to perform a study to assess what levels (if any) TCLP pollutants may be leached from representative cured and uncured products manufactured by RMA members.

TCLP tests were performed on the following RMA member products.

7 products from tire manufacturers
1 product from a roofing product manufacture
3 products from belt/hose manufacturers
3 products from molded product manufacturers
1 product from a gasket/sealant manufacturer
1 product from a manufacturer of printer rollers

For the study Radian utilized the list of chemicals and methods taken from the proposed June 13 TCLP regulations. Since pesticides and herbicides are not found in member products, these listed pollutants were not included in the scope of this study.

The TCLP listed chemicals studied in this program and their regulatory limits are shown below.

	Contaminant	Regulatory Level
Volatile Organics	Acrylonitrile	5.0 mg/L
	Benzene	0.07
	Bis(2-choroethyl)ether	0.05
	Carbon Disulfide	14.4
	Carbon Tetrachloride	0.07
	Chlorobenzene	1.4
	Chloroform	0.07
	1,2-Dichlorobenzene	4.3
	1,4-Dichlorobenzene	10.8
	1,2-Dichloroethane	0.40
	1,1-Dichloroethylene	0.10
	2,4-Dinitrotoluene	0.13
	Hexachlorobutadiene	0.72
	Isobutanol	36.0
	Methylene Chloride	8.6
	Methyl Ethyl Ketone	7.2
	1,1,1,2-Tetrachloroethane	10.0
	1,1,2,2-Tetrachloroethane	1.3
	Tetrachloroethylene	0.1
	Toluene	14.4
	1,1,1-Trichloroethane	30.0
	1,1,2-Trichloroethane	1.2
	Trichloroethylene	0.07
	Vinyl Chloride	0.05
Semivolatile Organics	o,m,p-Cresols (ea)	10.0
	Hexachlorobenzene	0.13
	Hexachloroethane	4.3
	Nitrobenzene	0.13
	Pentachlorophenol	3.6
	Phenol	14.4
	Pyridine	5.0
	2,3,4,6-Tetrachlorophenol	1.5
	2,4,5-Trichlorophenol	5.8
	2,4,6-Trichlorophenol	0.30
Metals	Arsenic	5.0
	Barium	100
	Cadmium	1.0
	Chromium	5.0
	Lead	5.0
	Mercury	0.20
	Selenium	1.0
	Silver	5.0

1.2.2 EP TOXICITY COMPARISON

Radian also compared the results of the TCLP analyses to the results of tests on selected RMA products using the current waste characterization protocol, EP Toxicity. Pesticides and herbicides were not included in the comparison.

1.2.3 TLCP CAGED MODIFICATION

A modification to the TCLP procedure was proposed by EPA in the Federal Register on May 24, 1989 which would allow certain categories of waste to undergo TCLP without being ground This would include most RMA member products.

Although TCLP originally had no provision for the pre-testing of physical integrity of a product before subjecting it to the leaching process, the EP Toxicity procedure does. This EP Toxicity test is referred to as the SIP (structural integrity test).

EPA recognized that TCLP should have a test similar to the structural integrity test and proposed to use a stainless steel cage in a glass bottle to contain whole, non-friable samples during the leaching process. ASTM is studying a cage made of plastic rather than stainless steel to avoid contamination by metals leached from the cage during the process.

The purpose of the cage is to shield the glass tumbler in which the whole sample is leached from hard, rock-like wastes. As initially proposed, TCLP required waste to be reduced so that it would pass through a 9.5 mm sieve.

Unlike vitrified or solidified wastes, the rubber products being studied for this project could not harm the tumbler when inserted as unground samples and therefore no caging apparatus was required.

1.3 PROJECT COORDINATION

Radian anticipated the need for confidentiality in this program and assigned codes to the participating manufacturers. The codes were assigned and maintained by Mr. Robert Richardson, the Radian manager for this project.

Radian provided participants with a detailed sampling protocol written to ensure that the samples provided to the laboratory were representative of the whole product and were not contaminated by the sampling process. To expedite prompt delivery, Radian also provided pre-labeled sampling containers to the participating manufacturers.

Initial processing of the samples to the appropriate (< 1 cm) size was done in Radian's Material Science Laboratory in Milwaukee, Wisconsin. This facility had appropriate hardware to grind and/or cut samples without contamination. All laboratory testing was performed at Radian's Austin facility. Data maintained at Radian are still coded. All laboratory operations conformed to EPA SW846 protocols for chain-of-custody, sample management, and laboratory analyses.

All analyses performed under EPA SW846 protocols applied the Third Edition methods and QC criteria.

2.0 RESULTS

2.1 PRODUCT CATEGORIES

The products contributed by RMA membership for the TCLP study included:

	NUMBER		NUMBER
TIRE PRODUCTS		MOLDED PRODUCTS	
Truck tires	1	Automotive Weather Strip	1
Light truck tires	2	Pipe Connecing Sleeve	1
Passenger Auto tires	4	Automotive Glassrun Channel	1
ROOFING PRODUCTS		SEALANTS	
Roofing - rubber sheet	1	Caliper Boot Seal	1
BELTS/HOSE		PRINTING ROLLS	
Automotive belts	2	Printer Roller	1
Automotive hoses	1		

2.2 RESULTS SUMMARY

With the exception of methyl ethyl ketone (MEK) detected in the printer roller product, values of all organics and metals were well below proposed regulatory values. The printer roller MEK value was 7.0 mg/L. The proposed regulatory limit is 7.2 mg/L.

Data also indicated no significant differences between the TCLP procedure as initially proposed on June 13, 1986 and the modified "caged" method proposed on May 24, 1988.

Finally, the data showed no consistant differences in levels of metals leached by the EP Toxicity procedure (cured and uncured samples) when compared to the TCLP extraction procedure.

2.3 DATA ANALYSIS

2.3.1 TCLP PROTOCOL ANALYSES FOR CURED SAMPLES

The concentrations of TCLP compounds detected in cured product samples leached and analyzed by the June 13, 1986 procedures are summarized in Table 1. For comparative purposes the proposed limits are provided at the heading of each column.

Although constituents listed by TCLP as hazardous compounds were found in all test categories, none exceeded proposed regulatory levels. Methyl ethyl ketone was detected in the printer roller sample at 7.0 mg/L. The proposed limit is 7.2 mg/L.

Most compounds were found at trace levels from ten to one hundred times less than TCLP regulatory limits. Many TCLP listed chemicals were not detected in any of the cured samples. These are listed below.

TCLP Listed Chemicals Not Detected* In Cured Samples

Metals

Silver

Volatile Organics

Acrylonitrile	1,2--Dichloroethane
Benzene	1,1-Dicholorethene
Carbon Tetrachloride	Isobutanol
Chlorobenzene	Methylene Chloride
Chloroform	1,1,1,2-Tetrachloroethane
1,1,2,2-Tetrachloroethane	Tetrachloroethylene
1,1,1-Trichloroethane	1,1,2-Trichloroethane
Trichloroethylene	Vinyl Chloride

Semivolatiles

This list continues following Table 1.

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* Not detected at or above method detection limits

TABLE 1.

TCLP PROTOCOL TEST – POSITIVE RESULTS

UNITS: mg/L

MDL = Method Detection Limits
a,f = TCLP cured

	VOLATILES			SEMI-VOLATILES	METALS						
SAMPLE ID	Carbon Disulfide	Methyl Ethyl Ketone	Toluene	Phenol	Arsenic	Barium	Cadmium	Chromium	Lead	Mercury	Selenium
MDL	0.005	0.1	0.005	0.01	0.001	0.01	0.005	0.01	0.002	0.0002	0.002
Regulatory Level	14.4	7.2	14.4	14.4	5	100	1	5	5	0.2	1
TIRE PRODUCTS											
1a	0.034	•	0.011	0.013	•	0.083	•	0.048	•	0.0002	•
2a	0.035	•	0.007	0.010	•	0.065	•	0.028	0.016	•	•
3a	0.067	0.021	0.050	•	•	0.150	•	0.012	0.009	•	•
4a	0.017	•	0.010	0.022	•	•	•	0.035	0.014	•	•
5a	•	•	0.190	0.046	•	0.570	•	0.037	0.002	0.0004	•
6a	•	•	•	0.045	•	0.590	•	0.025	0.002	•	•
7a	•	•	0.020	•	•	0.021	•	0.047	0.016	•	•
ROOFING PRODUCTS											
8a	0.160	•	0.007	•	•	0.045	•	•	0.014	•	•
BELTS/HOSE											
9a	0.520	•	0.038	1.30	•	0.590	•	•	0.005	•	•
9f	0.032	•	0.006	•	•	0.012	•	•	0.390	•	•
10a	0.790	•	0.240	0.064	0.007	0.012	0.006	•	0.003	•	0.003
MOLDED PRODUCTS											
11a	0.460	•	0.053	•	•	0.039	•	•	•	•	•
12a	0.320	•	0.074	•	•	0.540	•	•	0.007	•	•
13a	0.035	•	•	•	0.003	0.018	•	•	0.006	•	•
SEALANTS											
14a	•	•	•	•	0.004	0.018	•	0.030	0.036	•	•
PRINTING ROLLS											
15a	2.60	7.00	0.018	•	•	•	•	•	0.008	•	•

• These compounds were not detected or detected below method detection limits.

TCLP Listed Chemicals Not Detected* In Cured Samples, Cont.

Semivolatile Organics

Bis(2-chloroethyl)ether	o,m,p-Cresols
1,2-Dichlorobenzene	1,4-Dichlorobenzene
2,4--Dinitrotoluene	Hexachlorobenzene
Hexachlorobutadiene	Hexachloroethane
Nitrobenzene	Pentachlorophenol
Pyridine	2,3,4,6-Tetrachlorophenol
2,4,6-Trichlorophenol	2,4,5-Trichlorophenol

* Not detected at or above method detection limits

2.2.2. A COMPARISON OF CURED VS. UNCURED SAMPLES USING TCLP

Selected uncured samples representing each RMA product group also underwent TCLP leaching and analysis. Each uncured sample selected for the study had a matching cured product which also had undergone TCLP leaching and analysis. Two samples were selected from the tire products group and one sample was selected from each of the remaining product groups. The comparative cured and uncured sample results are shown in Table 2.

Note that the uncured printer roller sample values for methyl ethyl ketone (MEK) were 2.3 mg/L and were 7.0 mg/L for the cured sample.

As with the cured samples, the TCLP leachates of uncured samples did not exceed proposed regulatory limits. In comparing leachates from cured and uncured products, no consistent trend could be identified in the analysis results. For example, carbon disulfide values of cured samples exceeded uncured samples for two product categories and the profile was reversed for two other product categories.

TABLE 2.

TCLP PROTOCOL: A COMPARISON OF CURED AND UNCURED SAMPLES

UNITS: mg/L

MDL = METHOD DETECTION LIMIT
a = TCLP (cured)
b = TCLP (uncured)

	VOLATILES					SEMI-VOLATILES	METALS					
Sample ID	Carbon Disulfide	Methyl Ethyl Ketone	1,1,1-Trl Chloroethane	1,1,2,2-Tetra Chloroethane	Toluene	Phenol	Arsenic	Barium	Chromium	Lead	Mercury	Selenium
MDL	0.005	0.1	0.005	0.005	0.005	0.01	0.001	0.01	0.01	0.002	0.0002	0.002
Regulatory Level	14.4	7.2	30	1.3	14.4	14.4	5	100	5	5	0.2	1
						TIRE PRODUCTS						
3a	0.067	0.021	*	*	0.050	*	*	0.150	0.012	0.009	*	*
3b	0.012	*	0.005	*	0.017	*	*	0.072	0.023	0.008	*	*
5a	*	*	*	*	0.190	0.046	*	O.570	*	*	0.0004	*
5b	*	*	*	*	0.120	0.059	0.002	0.036	0.025	0.005	*	*
						ROOFING						
8a	0.160	*	*	*	0.007	*	*	0.045	*	0.014	*	*
8b	0.610	*	*	0.008	0.017	*	0.004	0.064	*	0.010	*	0.009
						BELTS/HOSES						
9a	0.520	*	*	*	0.038	1.30	*	0.590	*	0.005	*	*
9b	0.089	*	*	*	0.150	0.088	0.001	0.069	0.015	*	*	*
						MOLDED PRODUCTS						
13a	0.035	*	*	*	< *	*	0.003	0.018	*	0.006	*	*
13b	0.660	*	*	*	0.022	*	0.005	0.047	0.010	0.010	*	0.003
						SEALANTS						
14a	*	*	*	*	< *	*	0.004	0.018	0.030	0.036	*	*
14b	*	*	*	*	0.012	*	0.002	0.030	*	*	*	*
						PRINTING ROLLS						
15a	2.60	7.0	*	*	0.018	*	*	*	*	0.008	*	*
18b	1.20	2.3	*	*	0.017	0.012	0.001	0.080	*	0.032	*	*

* These compounds were not detected or detected below method detection limits

The list of TCLP compounds not found in the uncured samples was substantial and included:

TCLP Listed Chemicals Not Found In Uncured Samples

Metals	
Silver	Cadmium
Mercury	
Volatile Organics	
Acrylonitrile	Benzene
Carbon Tetrachloride	Chlorobenzene
Chloroform	1,2-Dichloroethane
1,1-Dicholorethene	Isobutanol
Methylene Chloride	1,1,1,2-Tetrachloroethane
Tetrachloroethylene	1,1,1-Trichloroethane
1,1,2-Trichloroethane	Trichloroethylene
Vinyl Chloride	
Semivolatile Organics	
Bis(2-chloroethyl)ether	o,m,p-Cresols
1,2-Dichlorobenzene	1,4-Dichlorobenzene
2,4-Dinitrotoluene	Hexachlorobenzene
Hexachlorobutadiene	Hexachloroethane
Nitrobenzene	Pentachlorophenol
Pyridine	2,3,4,6-Tetrachlorophenol
2,4,6-Trichlorophenol	2,4,5-Trichlorophenol

• Not detected at or above method detection limits

2.2.3 A COMPARISON OF TCLP GROUND VS. UNGROUND SAMPLES

The modification to TCLP proposed on May 24, 1988 will allow for TCLP leaching of unground samples. To test the effects of the proposed modification to TCLP, selected cured samples representing each RMA product group underwent TCLP leaching and analysis, but without the reduction of particle size below one centimeter.

An comparative assessment of volatiles was not performed. The proposed May 24 modification does not address TCLP Zero Head Space Extraction for volatiles. This extraction still requires

the sample to be of sufficiently small size to be placed in to a compressible piston. For volatile organics, the study of ground "unground" samples would duplicate of the initial TCLP Zero Head Space leaching process conducted for the ground samples.

For comparative purposes, each of the unground, cured samples selected for the study had a matching ground, cured product which had undergone TCLP. This set of products also matched the uncured products which had undergone TCLP assessment. The comparative results for the ground and unground samples are provided in Table 3.

As with the ground samples, only trace levels of TCLP contaminants were found in the unground samples. Comparative differences between ground and unground samples can most likely be assigned to variances in sample constituency or analysis methods, rather than differences in the efficiency of the ground or unground approaches to leaching.

The list of TCLP compounds not detected above method detection limits in the unground samples includes:

TCLP Listed Chemicals Not Detected In Unground Samples*

Metals	
Silver	Cadmium
Semivolatile Organics	
Bis(2-chloroethyl)ether	o,m,p-Cresols
1,2-Dichlorobenzene	1,4-Dichlorobenzene
2,4-Dinitrotoluene	Hexachlorobenzene
Hexachlorobutadiene	Hexachloroethane
Nitrobenzene	Pentachlorophenol
Pyridine	2,3,4,6-Tetrachlorophenol
2,4,6-Trichlorophenol	2,4,5-Trichlorophenol

* Not detected at or above method detection limits

TABLE 3.

TCLP PROTOCOL TEST
A COMPARISON OF GROUND AND UNGROUND SAMPLES
UNITS: mg/L

MDL = Method Detection Limit
a = TCLP (cured)
c = TCLP (unground, cured)

	SEMI-VOLATILES	METALS					
Sample ID	Phenol	Arsenic	Barium	Chromium	Lead	Mercury	Selenium
MDL	0.01	0.001	0.01	0.01	0.002	0.0002	0.002
Regulatory Level	14.4	5	100	5	5	0.2	1
TIRE PRODUCTS							
3a	*	*	0.150	0.012	0.009	*	*
3c	0.049	*	0.140	*	0.019	*	*
5a	0.046	*	0.570	0.037	*	0.0004	*
5c	0.059	*	0.029	*	*	*	*
ROOFING							
8a	*	*	0.045	*	0.014	*	*
8c	*	*	0.540	0.010	*	*	0.009
BELTS/HOSES							
9a	1.30	*	0.590	*	0.005	*	*
9c	0.580	*	0.089	*	*	*	*
MOLDED PRODUCTS							
13a	*	0.003	0.018	*	0.006	*	*
13c	*	0.004	0.031	*	0.008	*	0.003
SEALANTS							
14a	*	0.004	0.018	0.030	0.038	*	*
14c	*	*	0.530	*	*	*	*
PRINTING ROLLS							
15a	*	*	*	*	0.008	*	*
16c	*	*	0.600	*	*	*	*

*These compounds were not detected or were detected below method detection limits

2.2.4 A COMPARISON OF THE TCLP AND THE EP TOXICITY PROCEDURE

The results of these tests are provided in Table 4.

The EP Toxicity tests focused on metals, since the only organics in the EP Toxicity characterization procedure are chlorinated pesticides and phenoxychlorinated herbicides.

Silver, arsenic, mercury, and selenium were not found in the EP Toxicity leachates. The comparison of EP Toxicity leaches of cured and uncured products with the TCLP leachates of cured products showed very low (trace) values for all metals except barium in the sets. And barium values were less than one percent of regulatory limits. At these trace levels, no definitive trends between EP Tox cured, EP Tox uncured, and TCLP leachates could be isolated.

TABLE 4.

TCLP AND EP TOX METALS – COMPARING CURED AND UNCURED SAMPLES

UNITS: mg/L

MDL = METHOD DETECTION LIMIT
a = TCLP (cured)
d = EP Tox (cured)
e = EP Tox (uncured)

Sample ID	METALS					
	Arsenic	Barium	Chromium	Lead	Mercury	Selenium
MDL	0.001	0.010	0.01	0.002	0.0002	0.002
Regulatory Level	5	100	5	5	0.2	1
TIRE PRODUCTS						
3a	*	0.150	0.012	0.009	*	*
3d	*	0.073	*	0.016	*	*
3e	*	0.041	*	0.030	*	*
5a	*	0.570	0.037	0.002	0.0004	*
5d	*	*	*	0.005	*	0.002
5e	*	*	*	0.004	*	*
ROOFING						
8a	*	0.045	*	0.014	*	*
8d	*	*	*	0.007	*	*
8e	*	*	*	0.010	*	*
BELTS/HOSES						
9a	*	0.590	*	0.005	*	*
9d	*	*	*	0.003	*	*
9e	*	0.017	*	0.004	*	*
MOLDED PRODUCTS						
13a	0.003	0.018	*	0.006	*	*
13d	*	*	*	0.002	*	*
13e	*	*	*	0.005	*	*
SEALANTS						
14a	0.004	0.018	0.030	0.036	*	*
14d	*	*	*	*	*	*
14e	*	*	0.028	0.005	*	*
PRINTING ROLLS						
15a	*	*	*	0.008	*	*
15d	*	0.019	0.015	0.007	*	*
15e	*	0.013	*	0.037	*	*

* These compounds were not detected or detected below method detection limits.

3.0. TECHNICAL APPROACH

3.1 TCLP OVERVIEW

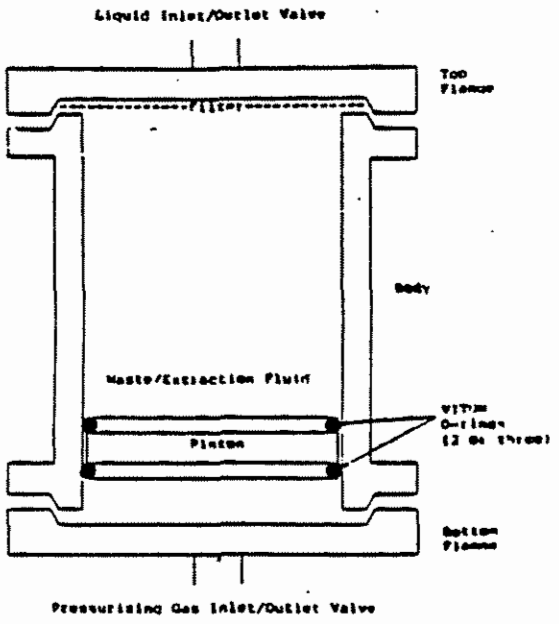
On June 13, 1986, EPA first proposed a rule intended to amend the waste characterization procedures to greatly expand the list of organic hazardous compounds by adding a volatiles extraction procedure (termed Zero Head Space) and by incorporating GCMS procedures for identification of both volatile and semivolatile organics. The proposed protocol was designated the Toxicity Characteristic Leaching Procedure or TCLP. Analytical methods used for both the EP Toxicity and the newer TCLP protocols remain those in EPA's RCRA analytical methods manual, SW846.

The purpose of TCLP, as well as the EP Toxicity protocol it was meant to replace, is to determine whether a waste has the potential to pose a significant hazard to human health or to the environment due to its propensity to leach toxic compounds into the groundwater.

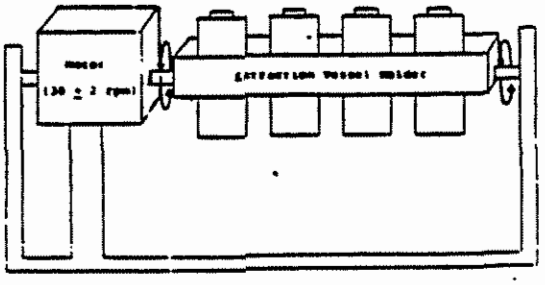
To leach semivolatile organics, pesticides, and metals the TCLP employs containment jar attached to a rotary tumbler spinning at a rate of 30 rmp. As noted above, the volatile organics are leached in a Zero Head Space apparatus, which is contains a self-enclosed piston system to force the leachate solution through a filter, effectively seperating the leaching medium from the sample without exposure to air. This apparatus is also attached to the rotary tumbler. The leaching solution for both processes is 0.1 M acetate buffer at a pH of 4.9 for non-alkaline wastes and pH 2.9 for alkaline wastes. Diagrams of both the Zero Head Space and Tumbler extractors are provided on the next page.

Other differences between the EP Toxicity procedure and TCLP included the extraction period (24 hours for EP Tox and 16 hours for TCLP) and initial abandonment of the EP Tox structural integrity procedure or SIP. As noted in the introduction, the EPA is seriously considering whether to reinstate some form of the SIP for monolithic wastes.

TCLP Extractors



Zero-Headspace Extraction Vessel



Rotary Tumbler

3.2 SAMPLING PROTOCOL FOR THE RMA PROGRAM

3.2.1 FIELD SAMPLING PROCEDURES

An example of the sampling instructions used in this project is provided as Appendix A. The sampling protocol written by Radian ensured both a coordinated effort among the participating companies and provision of samples representative of product constituents.

Radian provided participants with its SamplePak which contained collection and shipping instructions, chain-of-custody information, pre-cleaned containers, and prepared labels. The instruction set described above included use of containers, packing, security seal use, and shipping directions.

RMA participating companies provided samples of the products in sufficient quantity to provide TCLP ground and unground, TCLP uncured, and EP Tox analyses. The products analyzed included:

Item	Number Cured Product	Number Uncured Product TCLP Unground EP Toxicity
Tires	7	2
Belts/Hoses	3	1
Molded Products	3	1
Sealants	1	1
Roofing	1	1
Printer Rolls	1	1

3.2.2 LABORATORY SAMPLE PREPARATION

Samples were shipped by the participants to Radian's Material Science Laboratory in Milwaukee, Wisconsin for processing. Those portions identified for the standard TCLP analysis were chopped into portions of 1 cm or less. Care was

taken by Radian staff to use oil-free cutting tools and to avoid friction with the rubber material so as not to change its chemical make-up. The processed samples were then forwarded to Radian's Austin, Texas laboratory for leaching and chemical analysis.

3.2.3 SAMPLE CONTROL

Sample control for this program was coordinated through the use of a computerized laboratory data management system (SAM), which was developed by Radian. Sample log-in was performed immediately upon receipt of samples, and a unique sample code was assigned. SAM tracked the progress of samples through the laboratories, including such items as sample storage location, tests to be performed, holding/expiration dates, and reporting deadlines. Sample security arrangements and chain-of-custody procedures were all performed according to EPA Contract Laboratory Program guidelines using written SOPs.

The sample control center is equipped with locked, limited-access refrigerators (4°C) and freezer (-20°C) storage space. Samples remained in the sample control center storage until they were needed for preparation and analysis. After analysis unused samples and extracts were returned and retained by sample control.

3.3 TCLP LEACHING AND CHEMICAL ANALYSES

All chemical analyses were performed at Radian's Austin facility. Radian's Austin laboratory participates in the EPA WP (Water Pollution) and WS (Water Supply) performance evaluation programs and took the lead for Radian in the EPA TCLP evaluation program.

In providing an analytical approach to the TCLP analyses, absolute consistency with EPA quality assurance protocols were maintainted. This required that:

- A method blank be prepared for each TCLP and EP Tox extraction batch;
- The method of standard additions be used on all EP Tox metals analyses;
- Volatile and semivolatile matrix spike and matrix spike duplicates be run for batch;

Radian analyzed the RMA samples for TCLP constituents and EP Toxicity constituents following procedures outlined in (1) the proposed TCLP method printed in the Federal Register of June 13, 1986 and (2) Method SW1310 for EP Toxicity. Herbicides and pesticides were not included in this survey of compounds. Radian conformed to EPA SW846, Third Edition protocols for the laboratory analyses and QC criteria. Target compounds for this program were:

3.3.2 SAMPLE PREPARATION LABORATORY

Radian used twelve ZERO HEADSPACE extractors for TCLP volatiles extraction. Two of the extractors were used for method blanks, the other ten for actual analyses. In addition to zero headspace capacity, Radian also used rotary extractors sufficient for twenty simultaneous TCLP metals and semivolatile organic leaching operations. Radian's EP Toxicity leaching capability allows for the simultaneously leaching of 15 soils or wastes by EP Tox protocols.

The sample preparation laboratory consists of 3 rooms totaling 1500 square feet to provide 40 linear feet of hood space. With this hood space, the facility is equipped to run 50 continuous liquid-liquid extractions simultaneously. Each piece of equipment was thoroughly cleaned between each use to prevent contamination. Method blanks were used throughout the study to verify the integrity of these sample preparation procedures.

3.3.3 MASS SPECTORMETRY LABORATORY

Gas chromatography combined with mass spectrometry (GC/MS) employing EPA Methods SW8240 for the volatile compounds and SW8270 for the semivolatile constituents was used to identify the TCLP target chemicals. The characteristic retention times of compounds on a GC column provided presumptive evidence of their identity. This information in conjunction with mass spectra of the compounds obtained as they elute from the GC column yielded nearly unequivocal identification of the compounds.

The Austin GC/MS Laboratory occupies more than 1,200 square feet of laboratory space physically isolated from other Radian laboratories. The GC/MS lab maintains separate control of air temperature and humidity to prevent cross contamination of chemicals.

3.3.4 INORGANIC ANALYTICAL LABORATORY

Radian's Inorganic Analytical Laboratories provided analyses of metals, by atomic absorption spectrometers (three Perkin-Elmer 3030 AA and a Perkin-Elmer Model 403 AA), and Jarrel Ash 9000 and ARL 3400 Inductively Coupled Plasma Emission Spectrometer (ICPES). The two ICPES are capable of simultaneous analysis of the metals specified by EPA methods SW6010 and targeted by TCLP. The AAs were used for lead, mercury, and selenium analyses.

4.0 ADDITIONAL DATA

Radian Corporation is providing an associated document with this report which contains the following ancillary material:

1) Laboratory Reports - Includes analysis data, surrogate recoveries, matrix spike, matrix spike data, and preparation and anaysis histories;

2) Field Sample Information - Provides sample information and comments from the RMA participants; and

3) SW846 Methods and Proposed TCLP Protocols - Containing the EPA procedures for all tests performed as part of this study.

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APPENDIX A

An Example of A
Field Sampling Instruction Set for RMA Participants

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Radian Corporation SAMPLING PROTOCOL

Revision 1 - 5/30/89
RUBBER LEACHATE INVESTIGATION

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READ THESE INSTRUCTIONS CAREFULLY BEFORE COLLECTING SAMPLES.

QUESTIONS SHOULD BE DIRECTED TO

ROBERT RICHARDSON		PATRICK MEEHAN
PROGRAM MANAGER	or	CLIENT SERVICES REPRESENTATIVE
RADIAN CORPORATION		RADIAN CORPORATION
AUSTIN, TX		AUSTIN, TX

Telephone: 512 454-4797

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I. INTRODUCTION

The purpose of this program is to determine the extent, if any, to which rubber products leach hazardous constituents when placed in a landfill.

Radian Corporation will require representative samples of various rubber products to test for the materials' leaching characteristics. This protocol is designed to provide instructions for you, as the representative manufacturer, to collect and ship these samples to our laboratory.

Accompanying this protocol is a sampling kit with containers, address labels, chain of custody forms, and shipping instructions. A form has also been provided for sample information (product definition - such as the date produced, lot number, composition). For purposes of confidentiality, all information concerning participants and their samples have been assigned a numeric code. The Radian primary client code identifying RMA for this project is Vulcan. You will find this code and appropriate numeric assignments on your

chain-of-custody forms and sample labels. Do not write your company name or identify the RMA on the form, the container labels, or the address labels. Once the samples have been received at Radian, you will be contacted by the Client Services Coordinator to confirm sample identities and code assignments. The samples will then undergo EPA's Toxicity Characteristic Leaching Procedure (TCLP) and EPA's Extraction Procedure Toxicity Characteristic (EPTC or EP Tox). And finally, the leachates will be tested for organic and inorganic hazardous materials of interest to RMA membership.

II. SAMPLE INTEGRITY

Follow-on studies may be performed on the rubber products from which you are collecting these samples. Please store the products in an environment where they will not be exposed to oils or other chemicals, excessive humidity, temperature, or ozone for at least three months. RMA will provide guidance if longer term archival is required.

III. THE RADIAN SAMPLE KIT

The sampling kit consists of an insulated ice chest, one (1) prelabled 1-liter large mouth jar for each product sample undergoing an TCLP or an EPTC test, a prelabled chain-of-custody form (Attachment 1), packing materials, ice-packs (Blue Ice) for preservation during shipment, and prepared return address labels, and chain-of-custody seals to seal each container. You will also have a duplicate form for sample identification data (Attachment 2).

The 1-liter glass jar has been specially prepared to be free of organic materials. It is important that during handling, no object (including fingers) other than sample material be placed in the interior of the jar.

ATTACHMENT 1

RADIAN CORPORATION
8501 Mo-Pac Blvd.
P.O. Box 201088
Austin, TX 78720-1088

Chain of Custody Record

PROJECT

SITE

COLLECTOR

Analyses

SAMPLE I.D.	TYPE	DATE/TIME		REMARKS

RELINQUISHED BY:	DATE	TIME	RECEIVED BY:	RELINQUISHED BY:	DATE	TIME	RECEIVED BY:
RELINQUISHED BY:	DATE	TIME	RECEIVED BY:	RELINQUISHED BY:	DATE	TIME	RECEIVED BY:

RECEIVED FOR LABORATORY BY:	DATE	TIME	REMARKS

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ATTACHMENT 2 SAMPLE INFORMATION

CODE ASSIGNMENT: VULCAN-_____ COLLECTED BY: _____

DATE PRODUCT
WAS PRODUCED: _____ DATE COLLECTED: _____

A. SAMPLE IDENTIFICATION DATA:*

GENERAL DESCRIPTION (AUTOMOTIVE TIRE, BELT, ETC.) _____

DETAILED DESCRIPTION/NAME/MODEL NO: _____

LOT NUMBER OR OTHER UNIQUE IDENTIFIERS MARKINGS: _____

PRODUCT SIZE (VOLUME): _____ PRODUCT WEIGHT (LBS): _____

CHEMICAL COMPOSITION: _____

B. SAMPLE COLLECTION DATA:

BRIEFLY DESCRIBE YOUR PROCEDURES/APPROACH AND ANY DIFFICULTIES IN COLLECTING THE SAMPLE. _____

C. PROVIDE A SKETCH OF THE PRODUCT AND THE LOCATIONS FROM WHICH THE SAMPLE WAS COLLECTED. IF OTHER THAN A CROSS-SECTION WAS MADE, INDICATE WHY.

* We would appreciate your enclosing any additional (non-proprietary) printed material regarding this product's composition or usage, that is available.

Keep the jars closed until you are ready to add sample. Close them immediately after adding the sample.

For purposes of sample security, please retain a copy of the chain-of-custody form and the sample information form.

IV. INSTRUCTIONS

a) Lay out all the sample containers and supporting documentation. Each sample container is dedicated to a specific sample. If possible, about one liter volume of sample will be collected.

b) Freeze Radian provided Blue-Ice packs for 24 hours before use. If refrigeration is not available, secure a source of ice to be used in chilling the samples during shipment back to Radian. The ice should be carried in two ziplock bags, an inner bag and an outer bag. Leaking ice chests will be stopped by most shippers and the samples may become compromised. Several ice packs may be necessary to chill the samples.

c) Compare the coded sample labels with the code sheet provided in the sample kit. The labels will have a coded identification and the test to be performed. The code sheet should not be shipped back with the sample kit. It is for your reference. Radian program management will maintain two copies of the code sheet at their office.

d) Collecting representative samples - THIS IS A TEST OF THE PRODUCT...NOT JUST THE RUBBER MATERIAL IN THE PRODUCT...THEREFORE SAMPLES MUST BE COLLECTED WHICH ARE REPRESENTATIVE OF ALL OF THE PRODUCT'S CONSTITUENTS.

POWER TOOLS CREATE FRICTION WHICH CAN ALTER THE COMPOSITION OF THE SAMPLE. TAKE CARE TO KEEP HEAT AND CHEMICALS AWAY FROM THE PRODUCT BEING SAMPLED.

Instruction (e), below, provides information on general sampling size and weight requirements. For this program, it is extremely important that a sample be collected which is representative of the whole product. For homogenous samples or for regularly layered samples a cross section of the product will be representative.

For non-uniformly structured products samples must be collected which proportionately represent the entire product. For example, if a product has metal handles constituting 10% of the mass (by weight). Then 10% of the sample must be from the metal handles.

e) Collect the samples and fill the containers. If possible fill all containers to the neck of the bottle. For those samples designated for EPTC tests, attempt to provide material sized as large as possible (big pieces). Samples designated for TCLP will be shredded by Radian to less than 1 cm, therefore sample size is not a major consideration. At minimum we need the following sample sizes to allow for routine analysis as well as QC testing.

TCLP samples - 600 gms

EPTC samples - 300 gms

f) Keep the containers closed until you are ready to add the sample. After capping the containers, cover the neck and cap of each container with a custody seal in the fashion of the seal on a liquor bottle. Initial and date the custody seal. The custody seals will document tampering with the samples after collection.

g) Complete the chain-of-custody form by confirming the identity of each sample, adding the date and time of collection, the type of bottles, and the analyses to be performed. Conceivably, not enough sample may be available to fill the containers for all the analyses.

The chain-of-custody is an important document since it starts the chain of custody for the samples. Since this is a legal document, sign and date the form after completion.

Similarly fill out the Sample Information Form. Take care to provide all data which uniquely identifies the model and the product. Attach any other printed materials that may be available describing the product.

Keep a copy of the Sample Information form and the Chain-of-custody form. Send the original with the sample kit.

g) Carefully pack the samples in the ice chest for return to Radian. Separate the containers as well as possible within the chest and put packing material between them. Place sufficient ice or Blue Ice to keep the samples chilled during shipment.

h) Seal the ice chest with tape and place two custody seals across the lid and face of the ice chest. One seal should be across the front and a second seal on the side. Initial and date each seal.

i) Return the samples to Radian via an overnight delivery service (e.g., Federal Express). The samples should be sent to:

Radian Corporation
5101 West Beloit Rd
Milwaukee, WI 53214

ATTN: Chuck Applegate

Pre-addressed return labels will be included with the sample containers.

j) If questions arise concerning these instructions, shipment, preservation, or any other issue surrounding collection of the samples, contact:

Robert Richardson, (512)-454-4797, extension 5615 or

Pat Meehan, (512)-454-4797, extension 5159.