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Project Summary

Measurement of Volatile Organic Compound Capture Efficiency

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The U.S. Environmental Protection Agency (EPA) Office of Air Quality Planning and Standards (OAQPS) has issued new source performance standards regulating the emission of volatile organic compounds (VOC) from some surface coating operations. The regulatory compliance status in some cases requires determination of the overall VOC reduction efficiency based on a knowledge of the capture efficiency and control device efficiency. Presently, the only accepted method for determining capture efficiency, a gas-phase material balance, requires installation of an exhausted enclosure to collect and measure the fugitive VOC emissions.

The study reported here investigated alternate potential methods for determining capture efficiency which might not involve the expense and inconvenience associated with a temporary enclosure. Several approaches were considered, although the liquid/gas-phase material balance approach was selected for detailed testing. The liquid/gas-phase material balance approach was tested under laboratory and field conditions to evaluate the reliability of the available measurement techniques.

This Project Summary was developed by EPA's Hazardous Waste Engineering Research Laboratory, Cincinnati, OH, to announce key findings of the research project that is fully documented in a separate report of the same title (see Project Report ordering information at back).

Introduction

The EPA Office of Air Quality Planning and Standards (OAQPS) has issued New Source Performance Standards (NSPS) for some industrial surface coating operations using volatile organic compounds (VOC). Some operations employing solvent destruction systems must install exhausted total enclosures to demonstrate compliance through the measurement of capture efficiency and control device efficiency. The EPA Office of Research and Development in cooperation with the EPA OAQPS initiated the work described in the full report to explore more convenient and less costly alternatives for measuring capture efficiency.

The study was designed and conducted in two phases. Phase I was a review of existing information and recent studies and the development and feasibility evaluation of conceptual alternatives. Phase II was the detailed testing of the most feasible method determined from Phase I.

Alternate measurement concepts reviewed and considered included: material balance methods, tracer concepts, modelling, and indirect approximation techniques. The review included literature searches, conversations and site visits with plant representatives of surface coating operations potentially affected and review of previous material balance tests conducted at surface coating plants.

From the review, the liquid/gas-phase material balance and tracer gas concepts were found to be the most acceptable approaches. A comparison of the material balance and the tracer gas concepts concluded that both methods might be feasible and potentially applicable, but that the material balance approach is generally more acceptable. This conclusion was based on the premise that direct measurements are more readily accepted for compliance determination purposes than indirect determinations, and that the

material balance methods are further developed than tracer methods. Therefore, the liquid/gas-phase material balance methodology was selected for further testing and evaluation.

The liquid/gas-phase material balance approach compares the mass of gaseous VOC captured and sent to the control device with the mass of liquid VOC used (vaporized) in the process. Theoretically, the liquid/gas material balance approach has only one major disadvantage. That is, where drying ovens are direct fired, a portion of the captured VOC is destroyed making a liquid/gas mass balance technically infeasible.

The experimental testing of the liquid/gas-phase material balance method was conducted in two phases: laboratory testing and field testing. To properly evaluate the methodology, it was considered strategically important to begin testing under the simplest and most controlled conditions and to work up in complexity. The laboratory testing was a necessary step in this approach, since plant sites involve too many variables and potentially unmeasurable streams which would prevent a complete assessment of the measurement methods.

The objectives of the laboratory tests were to: evaluate the measurement methods for each required parameter, assess the overall ability to close liquid/gas-phase material balances under controlled conditions, and assess the impact of limited field test variables on the measurements. A systematic approach was taken in the laboratory tests in order to effectively evaluate the performance of the methodology. The test system was designed as a simple flow-through evaporation chamber, providing 100% capture and minimizing the number of measurement parameters. Therefore, a known capture efficiency value was established for comparison with the measured and calculated values, and sources of error in measurement were reduced to the lowest level. The only field test variables that were incorporated into the laboratory experimental design were those that were easily simulated and controlled and that might directly affect the statistical evaluation of the methodology.

In the laboratory tests, a known mass of liquid solvent or coating was placed in a heated evaporation pan located in the evaporation chamber and mounted on a balance. Air was pulled through the system at discrete flow rates to produce a mass flow of solvent laden air (SLA) through the measuring duct. While the known mass of liquid solvent was being evaporated, the resulting VOC concentration and the flow rate of the SLA stream were monitored and recorded for calculation of the gaseous VOC mass.

Twenty-four experimental runs were conducted while varying some of the test conditions in each run. The conditions were varied systematically through a fractional factorally designed test matrix in order to assess the impact of the common field test variables on the measurements. The variables included the composition of organics, the mass throughput of organics, and the gas stream flow rate. The vast majority of the laboratory tests involved pure solvents with no solids. However, in several tests commercial coating mixes containing solids were used.

Following the laboratory testing, a field test was conducted to evaluate the applicability of the laboratory tested methods in a field setting. The approach taken in testing involved selecting a test site with considerably more complex test conditions than the laboratory setting, but less complex than most coating operations. The selection criteria included a single coating line with a single applicator and near steady state operational conditions. The testing approach called for collecting continuous liquid and gas VOC data to enhance determination of capture efficiency over any given period. Sufficient data collection was also designed into the testing to allow an error analysis of the capture efficiency determinations.

The coating line tested was a magnetic tape coating process operating almost continually 24 hours per day. The liquid coating was pumped from the feed tank and applied directly to the web with no recirculation between the two points, except during splicing of the web rolls.

The test design required measurements similar to those in the laboratory test. The major difference between the field test and the laboratory test measurements was in the liquid-phase stream. The field test required determining the liquid mass through determination of the volume and density, since direct mass measurements could not be made.

Results and Conclusions

The laboratory mass balance experiments provided an opportunity to assess the ability to close liquid/gas-phase material balances using the available methodology, while conducting comparative testing of three different VOC analysis methods and testing of a method for the volatile content of the coating. Varied solvent amounts, solvent types, and gas flow rates were systematically introduced into the testing through a fractional factorally designed test matrix in order to simulate some important field variables. The test results for all experimental runs are shown in Table 1. Test results are also grouped by the gas-phase measurement method used in Table 2.

Material balance closures by Method 25A gas-phase measurements were by far the most successful in this set of experiments. Accuracy, bias, and precision were evaluated for the pure solvent and the commercial mixture experiments as separate groups. Accuracy seems more than adequate for each group since the mean closure values were, respectively, 99.9% (88.5-110%) and 102.2% (93.5-119%). Bias for either group was not statistically significant, since 100% was included within the 95% confidence interval in both cases. Precision, or variability, estimates for the two groups were excellent, since the coefficients of variation were 5.9 and 8.9%, respectively. Test results for the commercial coatings test runs were less accurate than pure solvent runs, probably due to the smaller change in liquid mass and lower SLA concentrations.

An analysis was conducted of the impact of test variables (e.g. mass throughput of organics, organic composition, and gas stream flow rate) on the Method 25A closure results. No variable was found to have any impact of practical significance.

Based on the laboratory test results, the liquid/gas-phase material balance method utilizing EPA Method 25A was further tested under field conditions. Capture efficiency determinations were made on an hourly basis during each test period using the gas-phase data collected from the total hydrocarbon analyzer and the liquid-phase data from the coating feed tank measurements. These determinations were made over a period of 114 hours, consisting of six discrete batches of liquid coating material.

Because the hourly capture efficiency determinations varied significantly, the data were further averaged over 24 hour periods and over the different batch periods to help smooth out the results and to narrow the confidence limits (see Table 3). The mean capture efficiency determination for five 24-hour periods was 106.7% with a coefficient of variation of 7.4%. The capture efficiency determina-

Table 1. *Liquid/Gas Mass Balance Closures*

Run No.	Elapsed Time[1] (min)	Solvent Type	Solvent Amount (liters)	Measured SLA Flow Rate[2] (SCFM)	THC Closures		Method 18 Closures			NIOSH 127 Closures		
					Aggregate[3] (%)	Total[4] (%)	MEK (%)	Toluene (%)	Total[5] (%)	MEK (%)	Toluene (%)	Total[5] (%)
1	100	MEK	2	832	100	100	73.0	—	73.0	397	—	397
2	56	Toluene	0.5	1400	101	101	—	98.0	98.0	—	210	210
3	140	Toluene	1	843	98.0	96.1	—	88.5	88.5	—	235[6]	235
4	61	Mixture[7]	1	1417	107	108	84.7	106	95.3	90.1	216	153
5	86	MEK	2	1825	88.5	88.5	103	—	103	117	—	117
6	51	MEK	1	1471	109	107	88.5	—	88.5	NM	—	NM[8]
7	53	Mixture[7]	0.5	1395	93.5	100	103	156	129	NM	NM	NM
8	150	Toluene	2	1406	98.0	99.0	—	119	119	—	NM	NM
9	70	Mixture[7]	0.5	844	90.1	89.3	75.8	90.1	82.9	69.0	155	112
10	70	Mixture[7]	1	1753	110	110	174	546	360	NM	NM	NM
11	105	Mixture[7]	1	918	102	101	78.7	97.1	87.9	123	204	163
12	35	MEK	0.5	1718	98.0	100	76.9	—	76.9	45.2	—	45.2
13	81	Mixture[7]	2	1461	102	100	84.7	95.2	89.9	131	350[6]	240
14	77	Toluene	1	1700	96.1	95.2	—	87.0	87.0	—	111	111
15	56	MEK	0.5	844	93.9	103	92.6	—	92.6	NM	—	NM
16	49	Mixture[7]	0.5	1787	99.4	100	96.1	95.2	95.6	50	149	99.5
17	60	Mixture[7]	2	1771	98.0	102	90.9	61.3	76.1	122	290	206
18	175	Mixture[7]	2	859	104	95.2	76.3	89.3	82.8	403	369	386
19	50	MEK	1	1834	100	107	77.5	—	77.5	NM	—	NM
26A	156	Comm 1[9]	1	1411	119	ND[10]	90.1	—	90.1	NM	—	NM
22	190	Comm 2[11]	1	1769	93.5	ND	117	—	117	NM	—	NM
23	121	Comm 2[11]	1	922	99.0	ND	86.2	—	86.2	118	—	118
26B	120	Comm 2[11]	1	1427	104	ND	98.0	—	98.0	NM	—	NM
31	241	Comm 3[12]	1	1730	99.0	ND	186	—	186	NM	—	NM

[1]*Elapsed time for each analysis varied due to cycle time and sensitivity differences.*
[2]*Flow rate measured by EPA Method 2 during each run. (SCFM 47.124 × feet per second.)*
[3]*Calculated from (MW × 100/CW) where MW = sum of the balance weight losses and CW = sum of the weight losses from the VOC concentration and the SLA flow rate. For the commercial mixtures, MW is the difference between the initial and final balance weights.*
[4]*Calculated from (TW × 100/CW) where TW is the initial total weight of solvent placed in the system.*
[5]*These data are the arithmetic average of the MEK and toluene closures.*
[6]*Average value of duplicate determinations.*
[7]*Mixture of 50% each by volume of MEK and toluene. Individual weights were used in the closure calculations.*
[8]*NM = not measured.*
[9]*Commercial mixture of rubber in MEK, specified by the manufacturer to contain 31% solids by weight.*
[10]*ND = not determined since all of the material was not evaporated.*
[11]*Mixture described in 9 above diluted approximately 10:1 with MEK.*
[12]*Mixture described in 9 above diluted approximately 2:1 with MEK.*

Table 2. *Analysis of Mass Balance Test Results*

Analysis Type	Data Type	Mean of Closures (%)	95% Confidence Interval (%-%)	Number of Observations	Standard Deviation (%)	Coefficient of Variation (%)
EPA Method 25A/Byron 401 THC Analysis	Pure solvent (overall)	99.9	97.1 - 102.7	19	5.9	5.9
	Commercial mix (overall)	102.2	89.6 - 114.9	5	9.1	8.9
EPA Method 18/GC-FID with Speciation	Pure solvent (overall)	105.4	75.0 - 135.9	19	63.9	60.0
	Commercial mix (overall)	115.5	64.4 - 166.7	5	41.2	35.7
NIOSH Method 127/Charcoal Tubes	Pure solvent (overall)	190.4	126.1 - 254.6	13	106.2	55.8

tions for six batch periods produced a mean of 103.0% and a 4.6% coefficient of variation.

In both cases above, the calculated mean capture efficiencies were higher than expected, since the maximum expected capture efficiency would be 100%. A review of the process streams measured, the measurement methods, and quality assurance results indicated no reason to believe that any source of VOC went unmeasured or that any measurement bias existed which would cause the higher measurement values.

In order to estimate the reliability of the capture efficiency results obtained in the field test, a complete error analysis was performed. Estimates for measurement errors were provided by an external audit or determined from repetitive measurements. The analysis showed that a single measurement has a 38% probability of being within ±5% of its true value. Finally, if 3 individual sets of closure measurements are made, the average value should be within ±10.7% of the true value with a 95% confidence limit. For six measurements, the average value would be within ±7.6%. Using the data collected at this site, it appears that the limits can only be narrowed to approximately ±4% of the true value.

The confidence interval would be expected to be much narrower for the same site using calculations based on a gas-phase material balance and an exhausted measurement enclosure. This is primarily due to the form of the gas-phase material balance equation. In the equation, the numerator and denominator are both composed primarily of the same measurement value, therefore, minimizing the impact of measurement bias or variability on the results.

Table 3. *Field Test Capture Efficiency Results*

Test Period	*Number of Tests*	*Range of Results*	*Mean of Results*	*Coefficient of Variation*
24 hours	*5*	*100.5 - 120.1%*	*106.7%*	*7.4%*
Coating Batch	*6*	*96.9 - 108.1%*	*103.0%*	*4.6%*

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Ronald J. Turner *is the EPA Project Officer (see below).*
The complete report, entitled "Measurement of Volatile Organic Compound Capture Efficiency," (Order No. PB 85-173 243/AS; Cost: $13.00, subject to change) will be available only from:
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