EVALUATION OF ATMOSPHERIC IMPACTS OF SELECTED COATINGS VOC EMISSIONS

Progress Report to the
California Air Resources Board
Contract No. 00-333

For the Period
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Summary of Progress

During the period of this report, progress was made in several areas but it was slower than expected because of problems that had to be resolved. Delays were encountered in getting the UCR EPA chamber facility completed and evaluated, though the final construction of the major components is nearly complete and characterization experiments should begin soon. Unfortunately, the EPA funding that was to be used to support the characterization experiments will run out in January, and at least some of the initial characterization work may need to be supported by this project until additional funding is obtained. The direct reactivity measurement system was modified to incorporate total carbon analysis, and although the initial version gave unsatisfactory results in the measurement of heaver VOCs, a version with redesigned combustion catalyst system appears to be satisfactory. This version is now being evaluated. Revisions were made to the analysis of the petroleum distillates studied by Censullo et al. since the submission of the first periodic report, and updates have already been provided to the CARB. A number of new VOC species were added to the SAPRC-99 mechanism as a result of emissions speciation work being carried out under separate funding, and these were incorporated in an update to the MIR scale that was provided at the request to the CARB. Since the SAPRC-99 mechanism has not undertone significant modification since it was documented in 2000, other than the addition of the new VOCs, the MIR update incorporates only a few changes to individual VOCs, due to correcting a few errors in the original tabulations, and some minor updates for individual types of VOCs. These areas are discussed in more detail below.

Progress in Preparation of the UCR EPA Chamber

The statement of work for this project calls for using the UCR EPA chamber for conducting environmental chamber experiments for selected coatings VOCs. Continuing progress was made, under EPA funding, to complete the construction of this facility in its final configuration so the needed characterization experiments could begin. The current status of this project, up to August 20, 2002, is given in an updated report to the EPA that was also provided to the CARB (Carter, 2002b).Briefly, the progress made in preparing this facility since the time of the last report for this project can be summarized as follows:

The design of the reactors and the associated frameworks and mixing systems were completed, and most of these components were constructed and evaluated. The design of the reactor support and portions of the mixing and exchange system is shown on Figure 1. Note that this shows only one of the two reactors that will be located side-by-side in the final configuration. The ~8’ x 8’ x 16’ FEP Teflon® film reactor is mounted to rigid upper and lower frames that eliminate all but one of the corner seals, simplifying reaction bag construction and reducing sources of leaks. The lower frame is mounted on the floor, through which the access and sampling panel and mixing and dump ports are attached. The upper frame is attached to the ceiling attached to a rotating shaft, which allows it to be raised or lowered as needed when the reactor is being filled and emptied. This arrangement allows the reactor to collapse as air is withdrawn for sampling, or for flushing or cleaning during experiments. Pressure controllers are used to maintain slightly positive pressure during experiments, with the top being lowered when the pressure falls below a set point due to sampling. Mixing is accomplished by rapidly pumping air through 8’’ ID Teflon® tubes using Teflon®-coated squirrel-cage blowers either to the other side of the reactor or the opposite reactor, with Teflon®-coated solenoid valves closing off the mixing system when not in use. Provision is made in the mixing system for injection of low volatility materials such as Texanol without excessive heating by integrating a low volatility VOC injection system into the chamber mixing system

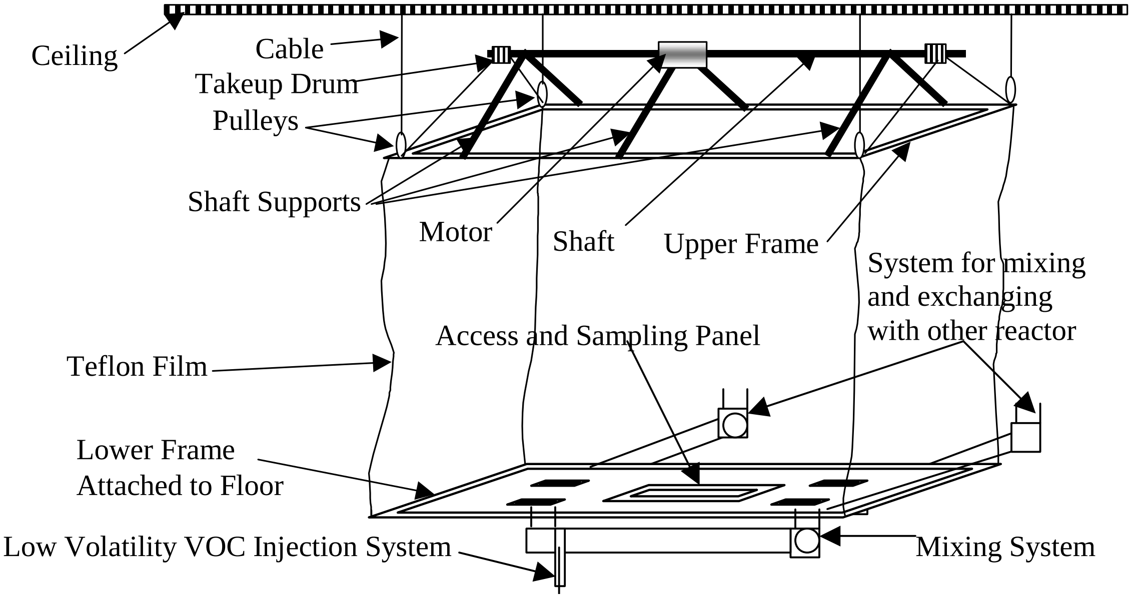

Figure 1. Design of the reactor support system and portions of the mixing and exchange system used for the UCR EPA chamber

The temporary pillowbag reactor discussed in the previous report was replaced with a new reactor in the configuration shown on Figure 1. This will serve as one of the two dual reactors to be used for this project. Installation of the second reactor was deferred until the performance of the first reactor in this configuration could be evaluated.

Tests with the new reactor indicated that leakage through seals or clamps in the Teflon film and in the mixing system were undetectable, but tests with CO introduced in the enclosure with pure air in the reactor indicated significant leakage around the flanges used for the access and sampling panels and mixing ports. This was eventually corrected by redesigning the flanges, but investigating and correcting the leaks caused delays in the program.

The spectral filter for the argon arc light source meeting the desired specifications was finally delivered, installed, and evaluated. The resulting spectrum is shown on Figure 2, where it is compared with that for ground-level sunlight. This spectrum meets our requirements, and is a somewhat better representation of the UV region than the Atlas Xenon arc system used in the XTC and CTC chambers (Carter et al, 1995).

Several preliminary experiments were conducted using the argon arc light source and the single reactor in the configurations discussed above. Leakage problems discussed above made the results of most not particularly useful for characterization. Some problems were encountered with excess electrode wear and other issues with the light source, but these are being resolved and should not cause delays in the future. The last experiments indicated that the major leakage and light problems have been resolved.

Once the flanges were improved, the performance of the first reactor appeared to be satisfactory. It was decided at this point to complete construction of the second reactor based on this design, so the system would be in its final configuration prior to characterization, and so two reactors could be irradiated at the same time, to improve productivity. Therefore, construction of the second reactor was

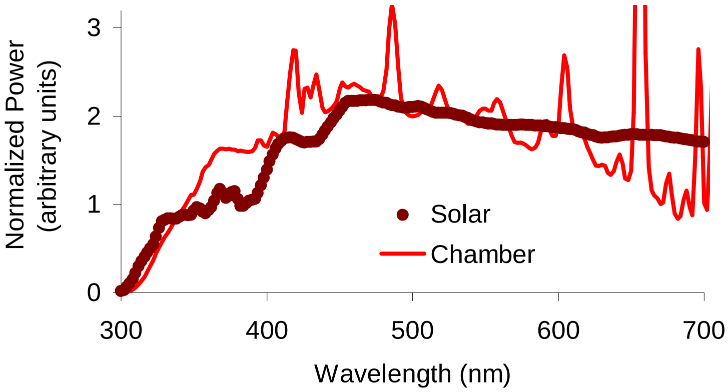

Figure 2. Relative power outputs of the light source for the UCR EPA chamber compared to solar output calculated for direct overhead sunlight.

begun, and other modifications were initiated to place the system in its final configuration. This process is underway now, but should be completed around the time this report is submitted.

Development of a Total Carbon Analysis Method for Use with Direct Reactivity Measurements

One of the objectives for this project is to adapt the direct reactivity measurement method developed under CARB contract 97-314 so it can be used for evaluating reactivities of major types of coatings VOCs, particularly Texanol and the various types petroleum distillates. As discussed in the final report for that project (Carter and Malkina, 2002), the GC methods currently used for determining how much VOC has been injected into the reactor are not satisfactory for evaluations of evaluation of complex mixtures or low volatility materials. Therefore, an initial priority for this project is to adapt a total carbon analysis method into this system. During the period of this report, the system discussed in the previous report, shown on Figure 3, was completed, the leakage problems were corrected, and exploratory experiments were carried out using propane, n-octane, n-decane, n-dodecane, n-tetradecane, n-hexadecane, and a mineral spirits sample "B" used in the Safety-Kleen study (Carter et al, 1997) as the test compounds. The system was reconfigured for reasons discussed below, and additional tests were then carried out with propane, n-dodecane, and n-tetradecane.

Figure 4 shows plots of measured vs. injected total carbon in the system configured as shown on Figure 3. Separate plots are shown for the two time periods because the response of the CO_2 analyzer apparently changed by about 30% during the time between May and September. The left hand plot shows that good agreement is obtained in total carbon measurements with propane, n-octane, and n-tetradecane when measured as CO_2 after passage through the combustion catalyst used in the first series of experiments. The data are well fit by a 1:1 line. Some time elapsed and the combustion catalyst was changed between the first and second series of runs, and plots of measured vs. injected total carbon with this system as interfaced to the system are shown on the right hand plot on Figure 4. It can be seen that essentially the same response is obtained with propane as with CO_2 itself, indicating essentially 100% conversion by the combustion catalyst and a reasonably precise total carbon measurement.

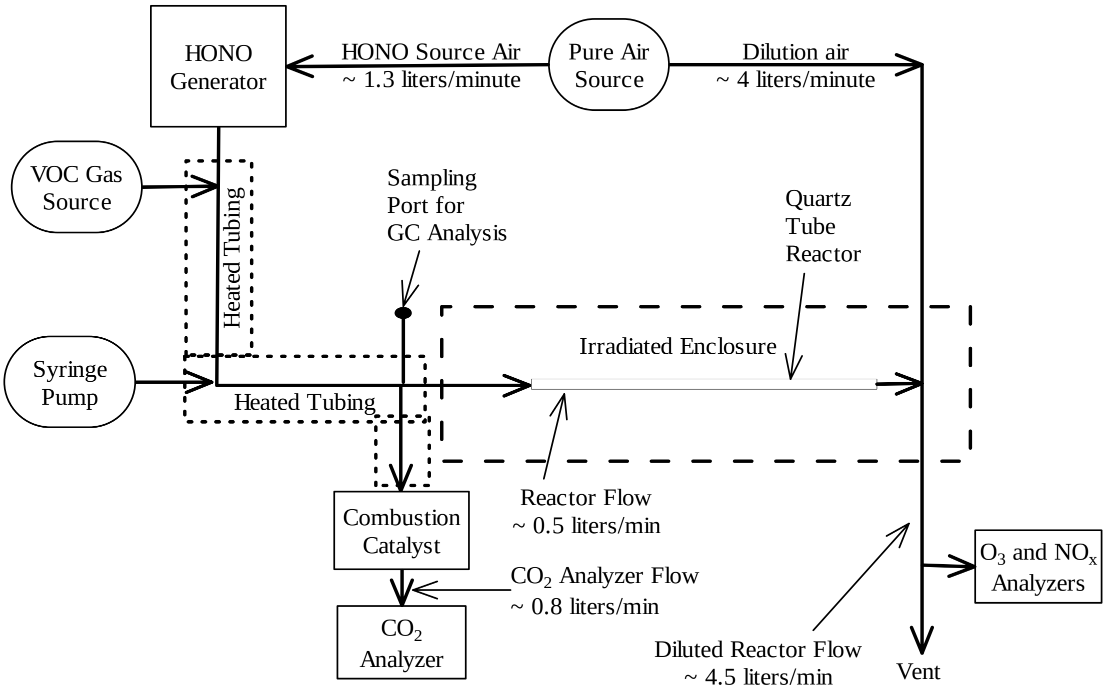

Figure 3. Diagram of setup for plug flow reactor being evaluated for use with the total carbon analysis system.

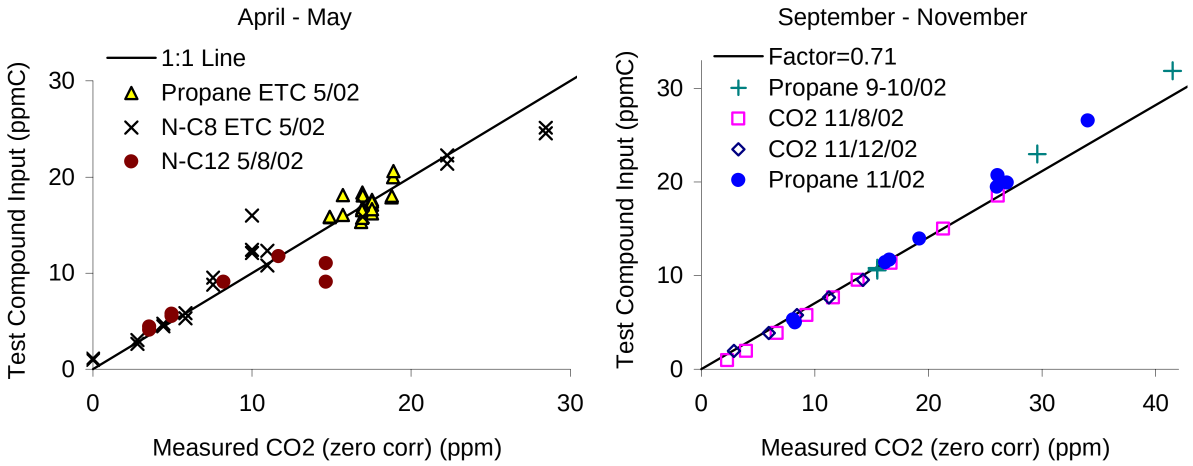

Figure 4. Plots of CO_2 or alkane carbons injected against CO_2 measurements when interfaced to the reactor system shown on Figure 3.

The tests in the first series of runs gave reasonably satisfactory results with propane and n-octane, but the data with the higher alkanes, including the mineral spirits itself, indicated a hysteresis effect in the direct reactivity response relative to the total carbon analysis. In particular, the reactivity response when the amount of VOC injected was increasing tended to be higher than that when the amount was decreasing, with the differences being the greatest when the rate of change was the highest. This is shown on Figure 5, which gives plots of the direct reactivity measure against CO_2 measured in that experiment. This can be explained by hang-up of the VOC in the system, specifically the combustion catalyst.

The combustion catalyst used in the first series of tests was taken from a Byron total carbon analyzer, where it was used for VOC removal purposes. The exact configuration of the Byron catalyst system was uncertain, as was the exact nature of the catalyst used. Because of this hang-up problem, it was replaced by a converter consisting of Hopcalite in a heated glass tube. It was found that this system gave much less hang-up with low volatility compounds if it is heated to ~570^{o}C. It was found that although 100% conversion to CO_2 occurs at lower catalyst temperatures, the hang-up is significantly reduced if a higher catalyst temperature is employed. This system was employed in the second series of tests, carried out beginning in September.

The system with the modified combustion catalyst gave results with significantly less hysteresis in experiments with heavier compounds. This can be seen in Figure 6, which gives results of experiments with n-tetradecane, whose carbon number is on the high end of the range for Mineral Spirits "B". Although there is some scatter in the reactivity results when plotted against the total carbon measurements, as indicated on the figure the outliers can all be associated with measurements made during upset or unstable conditions, as indicated by noise in the O_3 and NO data used to derive the reactivity measure. If the data are restricted to periods where stable readings are obtained, a good correspondence between the reactivity and carbon measurements are obtained, with the curve shape being as expected based on model calculations. Note in particular that there is no indication of different reactivity measurements being obtained when the VOC concentrations are increasing compared to when they are decreasing.

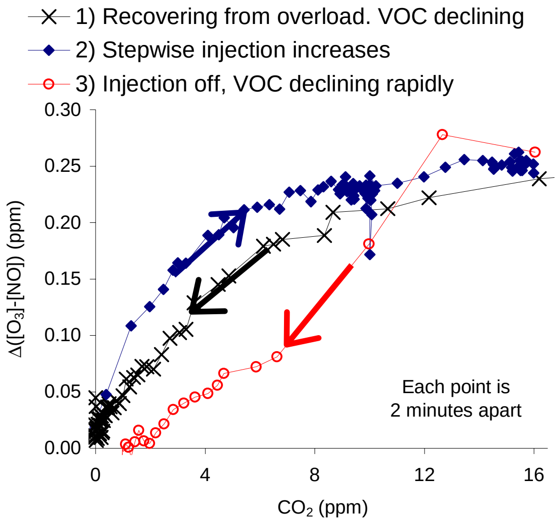

Figure 5. Plots of $\Delta([O_3]-[NO])$ vs. CO_2 in the mineral spirits "B" direct reactivity experiment carried out on May 24, 2002.

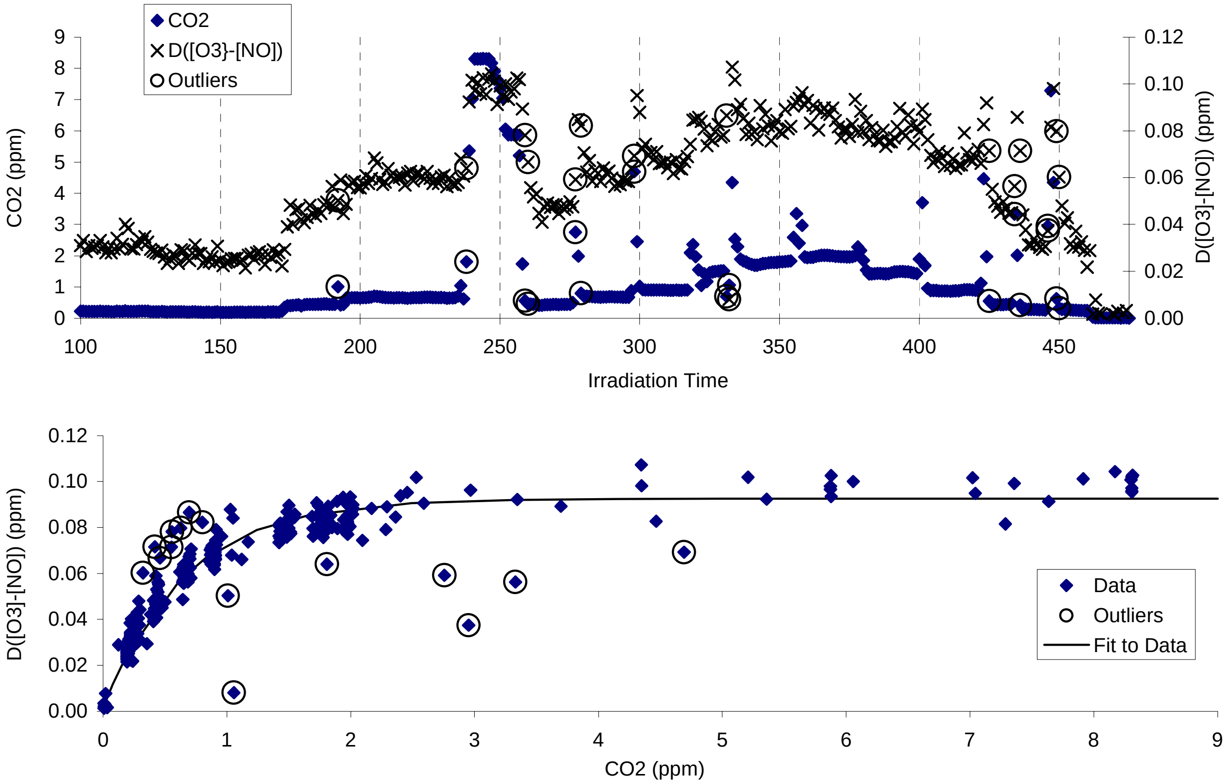

Figure 6. Plots of $\Delta([O_3]-[NO])$ and CO_2 vs. irradiation time and $\Delta([O_3]-[NO])$ vs. CO_2 in the n-tetradecane direct reactivity experiment carried out on October 17, 2002.

We are still in the process of characterizing the conditions of these experiments for modeling, and initial modeling of the experiments with the current setup tend to give consistently low predictions of the reactivity measure compared to the data. There are still some uncertainties in the flow measurements during the experiments, and the standard procedures for making these measurements are being still developed. We also need to obtain additional data for the system with the current setup and catalyst configuration.

Revisions to Solvent Analysis

The previous periodic progress report gave results of reactivity analyses of the solvents studied by Censullo et al (2002). The report used preliminary data provided by Censullo that contained some errors in boiling point ranges. Once the corrected data were available, and detailed compositional data for most of the solvents were also provided, the analysis was revised and was made available to the CARB and the Reactivity Research Advisory Committee members, and also posted on the project web site at http://www.cert.ucr.edu/~carter/coatings (Carter, 2002c). The results indicated that with the corrected boiling point ranges and the detailed compositional data the bin method developed by the CARB staff performed reasonably well in predicting the MIR's of the solvents studied by Censullo et al (2002).

Updated Reactivity Scale

At the request of the CARB staff, the Maximum Incremental Reactivity (MIR) values were recalculated for all organic compound categories currently represented in the SAPRC-99 chemical mechanism, and a spreadsheet file with the updated reactivity tabulation was provided to the CARB staff and made available at http://www.cert.ucr.edu/~carter/reactdat.htm.

Since neither the chemical mechanism nor the reactivity calculation scenarios or procedures had undergone any major revisions since the MIR values incorporated in the current aerosol coatings rule were calculated, MIRs changes were not expected for most of the species. However, some corrections had been made to some mechanism and emissions assignments for certain VOCs, and modifications or corrections have been made to some of the files and software programs. This resulted in resulting in non-negligible MIR changes for a small number of VOCs and very small changes for many others. Of the ~670 VOC categories on the CARB list for which MIRs were recalculated, the MIR's for 14 have changed by more than 10%, 26 have changed by more than 5%, and 305 have changed by more than 1%. In addition, in the process of making assignments for the SAPRC-99 mechanism for various emissions inventories and for other projects, ~95 of new VOC categories were added to the mechanism, and the MIRs for these new categories were calculated and added to the list. Table 1, lists the VOCs whose MIRs have changed by more than 5%, and Table 2 lists the new VOC categories that have been added to the current list. Footnotes to Table 1 indicate the reasons or probable reasons for the changes.

The current CARB tabulation includes MIRs for a number of species that were derived by CARB staff based on the upper limit estimation method. No updates had been made to the recommended procedures that would affect these upper limit MIR estimates, so no changes were made to these upper limit estimates. In addition, as indicated on Table 1, a few VOC categories for which the CARB made upper limit estimates are included on the current tabulation with lower estimated MIR values. Note that the estimates of the uncertainties for the reactivities of these compounds have not been reduced, so it is recommend that the CARB continue to use the upper limit estimates for those compounds for regulatory applications where this approach is appropriate.

Future Schedule

During the upcoming period, we hope to complete the evaluation and adaptation of the direct reactivity measurement method for application to reactivity analysis for petroleum distillates and other coatings VOCs, so it can be used to provide data relevant to this program. However, further work on this approach may be abandoned if new problems are encountered that indicate that further research is needed to make this method useful. This is because the funds budgeted for this task are insufficient for further development or evaluation, and as discussed below the priorities need to shift to the chamber studies. This will be discussed with the RRAC before any final decision is made. However, we are reasonably optimistic with the recent results with the total carbon analysis method that the method as currently developed will be useful for this project.

Barring additional unforeseen delays, the construction of the UCR EPA chamber should be completed before the end of November, and the initial characterization experiments with the chamber in the final, dual-reactor configuration should begin prior to the end of 2002. The initial plan for this project was to use the EPA funds to fully characterize the UCR EPA chamber and conduct systematic mechanism evaluation experiments with simpler chemical systems prior to utilizing it for studies of Texanol® and other coatings VOCs for this project. Unfortunately, the funding for the EPA project will be essentially exhausted in January 2003, and although we may obtain additional Federal funding later in the year, this CARB program is currently the only other project with funds to support the facility during the coming quarter. Therefore, at least some of the experiments for this project will need to be scheduled much earlier than initially anticipated, though obviously a minimum set of characterization experiments needed for using the data for mechanism evaluation will need to be conducted first. Since some characterization has already been carried out, this probably can be completed in about a month of full time operation.

Although we know we will be conducting experiments with Texanol®, because of the anticipated difficulties in handling and analyzing this low volatility compound, it is probably best that this not be the first compound studied in the new facility. We recommend that a relatively light petroleum distillate, such as those studied for Safety-Kleen (Carter et al, 1997), be studied first. It is expected that the choices for petroleum distillates for initial study will be selected at the upcoming RRAC teleconference, scheduled for December 4. Once the selection is made and samples are acquired, we will conduct initial experiments using a smaller chamber, and develop appropriate procedures for injection and analysis.

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Table 1. Listing of VOC species or categories whose MIR values have changed by more than 5%.

Model Name	Description	MIR (gm O_3 /gm VOC)			Note
		Updated	Previous	Change	
CL2IBUTE	2-(chloromethyl)-3-chloropropene	3.13	1.13	177%	[a]
AMP	2-Amino-2-Methyl-1-Propanol	4.75	15.08	-68%	[b]
TM-AMINE	Trimethyl Amine	7.06	16.60	-57%	[b]
244M2C5E	2,4,4-trimethyl-2-Pentene	8.52	5.85	46%	[c]
METHANE	Methane	0.014	0.01	39%	[d]
CYC-C15	C15 Cycloalkanes	0.65	0.99	-34%	[e]
PROPACID	Propionic Acid	0.79	1.16	-32%	[f]
ACETACID	Acetic Acid	0.50	0.71	-30%	[f]
2ETHXACD	2-Ethyl Hexanoic Acid	3.49	4.41	-21%	[f]
ME-BR	Methyl Bromide	0.017	0.02	-16%	[d]
CH3-CL	Methyl Chloride	0.034	0.03	15%	[d]
CHCL3	Chloroform	0.034	0.03	14%	[d]
ETOX	Ethylene Oxide	0.044	0.05	-11%	[d]
DPGOME2	2-[2-methoxypropoxy]-1-propanol	2.70	3.02	-11%	[g]
C7-OLE1	C7 Terminal Alkenes	4.20	4.56	-8%	[h]
1-HEPTEN	1-Heptene	4.20	4.56	-8%	[h]
11BR2-C2	1,2-Dibromoethane	0.046	0.05	-7%	[d]
C15-OLE1	C15 Terminal Alkenes	1.27	1.37	-7%	[h]
1-C15E	1-Pentadecene	1.27	1.37	-7%	[h]
ME-FORM	Methyl Formate	0.064	0.06	6%	[h]
DGBE	2-(2-Butoxyethoxy)-Ethanol	2.87	2.70	6%	[h]
CL2-ME	Dichloromethane	0.066	0.07	-6%	[d]
N-C18	n-C18	0.44	0.47	-6%	[i]
48DM-C14	4,8-Dimethyl Tetradecane	0.55	0.58	-5%	[i]
N-C15	n-Pentadecane	0.53	0.56	-5%	[i]

[a] The representation of the mechanism of this compound was changed. However, the mechanism is highly uncertain and the upper limit MIR probably should be used.

[b] The "previous" value is the CARB staff's estimated upper limit MIR. The upper limit value should continue to be used in the regulation for consistency with the current policy for treatment of uncertainty.

[c] The structure for this compound was incorrectly specified as that for 2,4,4-trimethyl-2-hexene when calculated previously.

[d] Change due to round-off error, since the CARB tabulation had only two significant figures. The MIR's are the same to two significant figures.

[e] The reason for this change could not be determined. This compound is represented using the lumped molecule method, and the calculated MIRs for the species used to represent it have not changed significantly.

[f] The estimated mechanisms for organic acids have changed due to a modification in the estimation of branching ratios for the initial OH reaction at various positions around carboxylic acid groups.

[g] Assignments for this compound could not be found on the previous databases.

[h] The change for C_7 or the C_{15} terminal alkenes is due to the change for 1-heptene or 1-pentadecene, respectively. The change for these higher molecular weight 1-alkenes must be due to some small change in the base mechanism or scenario assignments because the mechanism and the rate constants used are the same.

[i] The reactivities of these compounds are expected to be highly sensitive to small changes in the base mechanism or scenario conditions. The mechanisms for these compounds were not changed.

Table 2. Listing of VOC compounds and categories that have been added to the list.

Model Name	Description	MIR (gm O_3 / gm VOC)
BCYC-C8	C8 Bicycloalkanes	1.75
13E5PCC6	1,3-Diethyl-5-Propyl Cyclohexane	0.96
C3M2-C5E	Cis-3-Methyl-2-Pentene	12.84
M-ET-TOL	m-Ethyl Toluene	9.37
P-ET-TOL	p-Ethyl Toluene	3.75
O-ET-TOL	o-Ethyl Toluene	6.61
O-DE-BEN	o-Diethyl Benzene	6.61
M-DE-BEN	m-Diethyl Benzene	9.37
P-DE-BEN	p-Diethyl Benzene	3.75
1235MBEN	1,2,3,5 Tetramethyl Benzene	8.25
INDENE	Indene	3.21
ME-INDAN	Methyl Indans	2.83
C12-TET	C12 Tetralin or Indane	2.33
IAMOH	isoamyl alcohol (3-methyl-1-butanol)	2.66
MIBUCBN	4-methyl-2-pentanol (methyl isobutyl carbinol)	2.43
23M1C5OL	dimethylpentanol (2,3-dimethyl-1-pentanol)	2.45
1M1C7OL	5-methyl-1-heptanol	1.84
TMCYC6OH	trimethylcyclohexanol	1.88
26M2C7OH	dimethylheptanol (2,6-dimethyl-2-heptanol)	1.04
26M4C9L	2,6-dimethyl-4-heptanol	2.06
MENTHOL	menthol	1.62
1-C10-OH	1-decanol	1.19
37M1C8L	3,7-dimethyl-1-octanol	1.34
268M4C9L	Trimethylnonanolthreoerythro; 2,6,8-Trimethyl-4-nonanol	1.39
14-C4-OH	1,4-butanediol	3.04
PNTAERYT	pentaerythritol	2.23
2E13HXOL	2-Ethyl-1,3-hexanediol	2.15
13DXOLAN	1,3-dioxolane	5.50
14DXANE	1,4-dioxane	2.83
IPROIPR	diisopropyl ether	3.56
EGDEE	ethylene glycol diethyl ether; 1,2-diethoxyethane	3.13
ACETAL	acetal (1,1-diethoxyethane)	3.73
44MHX3O	4,4-Dimethyl-3-oxahexane	2.03
2M12M1MP	2-methoxy-1-(2-methoxy-1-methylethoxy)-propane	2.41
3MEOC3OH	3-methoxy-1-propanol	4.00
TH2FURM	tetrahydro-2-furanmethanol	3.49
PROC3OH	n-propoxypropanol	3.97
TEGLYCL	triethylene glycol	3.51
DPGEE	dipropylene glycol ethyl ether	2.88
TETGLCL	tetraethylene glycol	2.71
1BOEO2PR	1-(butoxyethoxy)-2-propanol	2.28
DBNP	glycol ether dpnb {1-(2-butoxy-1-methylethoxy)-2-propanol}	2.16
GBUTYACT	gamma- butyrolactone	1.10
IPRFORM	Isopropyl Formate	0.42
IAMACET	isoamyl acetate (3-methylbutyl acetate)	1.25
2M1BACET	2-methyl-1-butyl acetate	1.17
MAMACET	methyl amyl acetate (4-methyl-2-pentanol acetate)	1.50
5MC7-ACT	5-Methylheptyl Aceate	0.73
MELAURAT	methyl dodecanoate {methyl laurate}	0.52

Table 2 (continued)

Model Name	Description	MIR (gm O_3 / gm VOC)
ME-MYRST	methyl myristate {methyl tetradecanoate}	0.47
MEOPRACT	methoxypropanol acetate	2.09
GLY-ACET	glyceryl triacetate	0.57
DIPRADP	diisopropyl adipate	1.34
IBUTACD	isobutyric acid	1.28
BUTACD	butanoic acid	1.95
MALACD	malic acid	8.74
3MBUTAC	3-Methylbutanoic acid	4.80
ADIPACD	adipic acid	3.11
HOPRACR	hydroxypropyl acrylate	4.78
NBUACRAT	n-butyl acrylate	4.86
IBU-ACRT	isobutyl acrylate	4.58
ATRPNEOL	a-terpineol	5.04
2MEXAL	2-methyl-hexanal	3.95
MIPRK	Methyl Isopropyl Ketone	1.62
24C5-K	2,4-pentanedione	1.02
2PRCC6K	2-propyl cyclohexanone	1.59
4PRCC6K	4-propyl cyclohexanone	2.08
2M2C5E4O	mesityl oxide (2-methyl-2-penten-4-one)	17.40
ISOPRON	isophorone {3,5,5-trimethyl-2-cyclohexenone}	10.58
1C9E4ONE	1-nonene-4-one	3.19
DOHACT	dihydroxyacetone	3.89
C8-PHEN	C8 Alkyl Phenols	2.07
C9-PHEN	C9 Alkyl Phenols	1.86
C10-PHEN	C10 Alkyl Phenols	1.68
C11-PHEN	C11 Alkyl Phenols	1.54
C12-PHEN	C12 Alkyl Phenols	1.42
C6-ALK	Unspeciated C6 Alkanes	1.48
C7-ALK	Unspeciated C7 Alkanes	1.79
C8-ALK	Unspeciated C8 Alkanes	1.64
C9-ALK	Unspeciated C9 Alkanes	2.13
C10-ALK	Unspeciated C10 Alkanes	1.16
C11-ALK	Unspeciated C11 Alkanes	0.90
C12-ALK	Unspeciated C12 Alkanes	0.81
C13-ALK	Unspeciated C13 Alkanes	0.73
C14-ALK	Unspeciated C14 Alkanes	0.67
C15-ALK	Unspeciated C15 Alkanes	0.61
C16-ALK	Unspeciated C16 Alkanes	0.55
C17-ALK	Unspeciated C17 Alkanes	0.52
C18-ALK	Unspeciated C18 Alkanes	0.49
C10-ARO	Unspeciated C10 Aromatics	5.48
C11-ARO	Unspeciated C11 Aromatics	4.96
C12-ARO	Unspeciated C12 Aromatics	4.53
MS-802	Composite mineral spirit (CARB Profile ID 802)	2.02
ISOPARM	Exxon Isopar(r) M Fluid	0.65
MS-CP96	Thinning Solvent/Mineral Spirits (Cal Poly Slo. 1996)	1.99