



PECKSR

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To: SZAKATBK, MILLERSR, HALBERRM
cc: PLAYTIS, MILLERW, PARSONMA, MAYLESM, ZIPFEL
Subject: pathforward and problems with determination of C8 in air

Based on high reported C8 results for the air samples collected on 7/24/98, and due to erratic GC results, I recommend resamples be taken from these locations. If possible, duplicate samples should be collected. This will help ensure accurate results and allow for some analytical development as described below.

Several problems were observed in the analysis of the samples. Most significantly, there was an abnormally large variation in the response of the C10 internal standard (42%) which is not understood. This certainly influenced calculation of the results reported 7/25 since the C8 response is normalized for C10. Additionally, the linearity of the calibration plot ($R^2=0.908$) was very poor (a much more linear response was seen if the correction for C10 was ignored). Another abnormality was the samples were mistakenly left in the hot block for an excessive time; the impact of this is unknown.

The calibration was repeated on 7/27 with excellent results. Variation in C10 peak heights was less than 4%. A series of standards without added C10 was also analyzed, and the GC response was extremely linear with C8 ($R^2=0.995$).

In the table below, C8 results from different calibration plots are shown. Results above the AEL of 0.5 mpb are in bold. Different calculations are as follows:

1. The first column ("w/C10") shows the original results calculated per the method where the C8 peak heights are corrected (normalized) based on the C10 peaks. Calibration samples from 7/24.
2. The second column of results were based on the uncorrected C8 peak heights using the calibration samples which were analyzed on 7/24.
3. The third column of results were based on the uncorrected C8 peak heights using the calibration samples which were analyzed on 7/27.
4. The fourth column of results ("no C10") were based on a series of calibration samples which were prepared without C10.

Table: Calculated C8 in Air (moles per billion)

SAMPLE	LOCATION	ignore C10		no C10	
		w/C10	7/24 cal	7/27 cal	7/27 cal
A	smoking area; 162 roof	0.710	0	0.195	0.303

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B	blank	0.456	0.141	0.407	0.523
C	top of SW stairs, unit side	0.859	0.420	0.763	0.863
D	SE rail	0.808	0	0.120	0.252
E	mid-rail, North	0.152	0	0.170	0.282

Several comments are in order:

- o Column 1 (original) results are invalid due to excessive variation in C10 response.
- o Column 2 results are significantly lower than column 3 due to differences in the slopes and offsets of the 2 calibration plots.
- o Calibration plots for column 2 and 3 results showed good linearity ($R^2=0.984-0.985$) even though the impact of the added C10 (which contains some C8 impurity) was ignored.
- o Results in the fourth column are higher since the standards were prepared without C10. The air samples contained extra C8 (from the C10 impurity) which was not subtracted in this calculation.
- o Standards prepared on the same day (7/27) showed quite similar calibration plots.
- o Response for standards prepared on different days (7/23 vs 7/27) was quite different.

In this series of samples, the C10 added standard seriously complicated the calculations. We observed linear GC response with NO added C10 (a "classical" source of variation, injected volume, has effectively been eliminated with the use of autosamplers). I am hoping to demonstrate that the method can give reliable determination of C8 in air when no C10 is added. This would streamline the assay and allow more rapid turnaround. We are requesting duplicate air samples so one set can be analyzed w/o C10.

One area that still concerns me is the day-to-day variation in GC response for the standards. Analysis of standards is being performed today in duplicate. This will give more information to help quantify day-to-day variance as well as ensuring quality of reagents for use with additional air samples.

Comments are welcome.

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