

Accuracy of Vapor Dosing with the AutoChem

The Vapor Generator option for the AutoChem provides a means to quantitatively inject pulsed dose amounts of vapor onto a solid sample. Vapors of most interest are actually gases below their critical temperature; hence, they are condensable. Design features and operation of the AutoChem's vapor generator are described in Product Bulletin 83.

Reagent grade 2-propanol (boiling point 97 °C) was used to demonstrate the accuracy and reproducibility of the AutoChem and vapor generator for dosing vapor at a specific pressure. A series of three experiments was performed in which 15 repeated pulses of 2-propanol were made from the vapor generator with an inert helium carrier gas. The three different vapor pressures were obtained using reflux zone temperatures of 60, 70, and 80 °C.

The conditions of the experiments were:

- An empty sample tube; the furnace was maintained at 120 °C to prevent condensation of the alcohol in the tube.
- All compartments of the AutoChem were heated to 130 °C.
- Helium was used as the loop gas and the carrier/reference gas.
- The boil-zone temperature of the vapor generator was held 20 °C higher than the reflux zone.
- A nominal 0.5-cc injection loop was employed. The exact volume of the loop at 130 °C was 0.3363 cc.

Tables I, II and III show the integrated peak areas for all 15 injections at the three respective vapor reflux temperatures of 60, 70, and 80 °C.

Table I. Eluted peak areas for 15 sequential loop injections of 2-propanol vapor using a reflux temperature of 60 °C and a boil temperature of 80 °C.

Peak Number	Area	Peak Number	Area	Peak Number	Area
1	-0.0371	6	-0.0342	11	-0.0340
2	-0.0342	7	-0.0341	12	-0.0342
3	-0.0342	8	-0.0340	13	-0.0342
4	-0.0343	9	-0.0341	14	-0.0341
5	-0.0344	10	-0.0340	15	-0.0341

Table II. Eluted peak areas for 15 sequential loop injections of 2-propanol vapor using a reflux temperature of 70 °C and a boil temperature of 90 °C.

Peak Number	Area	Peak Number	Area	Peak Number	Area
1	-0.0531	6	-0.0518	11	-0.0528
2	-0.0519	7	-0.0512	12	-0.0522
3	-0.0522	8	-0.0511	13	-0.0517
4	-0.0516	9	-0.0517	14	-0.0520
5	-0.0521	10	-0.0517	15	-0.0520

Table III. Eluted peak areas for 15 sequential loop injections of 2-propanol using a vapor reflux temperature of 80 °C and a boil temperature of 100 °C.

Peak Number	Area	Peak Number	Area	Peak Number	Area
1	-0.0877	6	-0.0860	11	-0.0866
2	-0.0866	7	-0.0866	12	-0.0854
3	-0.0867	8	-0.0867	13	-0.0853
4	-0.0866	9	-0.0857	14	-0.0848
5	-0.0867	10	-0.0854	15	-0.0853

The three TCD traces are shown overlaid in Figure 1.

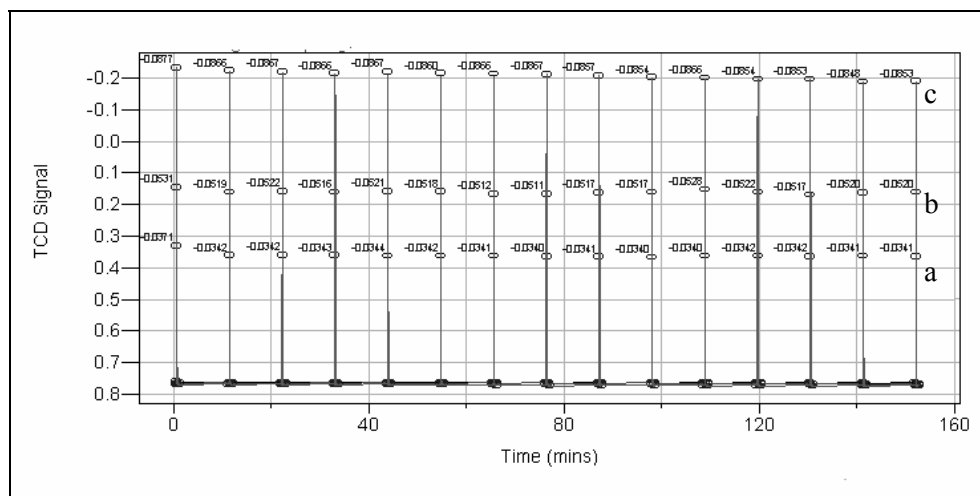


Figure 1. The time vs. signal outputs from the TCD detector for 15 sequential injections of 2-propanol vapor at three different reflux temperatures; (a) 60 °C, (b) 70 °C, and (c) 80 °C.

The summary data are given in Table IV. The standard deviations for the integrated peak areas for the three different temperature series are all well below 1%. The active volume injected compares quite well with that expected from the known vapor pressure of the 2-propanol.

Table IV. Comparison of the vapor pressure of 2-propanol to the average peak areas obtained, including the calculated standard deviation, and compared to the calculated vapor volumes injected.

Reflux Temperature (°C)	Vapor Pressure (torr)	Average Peak Area	Vapor Volume Injected (cc)	Standard Deviation of Area
60	140	0.03435	0.0642	0.00077
70	220	0.05194	0.0999	0.00052
80	360	0.08618	0.1636	0.00078