Lotus Consulting presents:

Gas Chromatography

PFPD TUNING APPLICATION
by Lotus Consulting

Sulfur-containing compounds have a very useful property of emitting a characteristic wavelength (465 nm) when sulfur-containing compounds are broken down in a flame and then thermally exciting the resulting sulfur dimer - S_2. This principle has been the basis for conventional flame photometric detectors in use with gas chromatographs for well over thirty years.

Despite the selectivity generated by filtering out only light from this emission, other species can generate an emission in the same wavelength region. Hydrocarbons passing through to this detector are also combusted and their products yield a chemiluminescence that coincides with the sulfur emission. This interference often yields false chromatographic peaks that could be quantified improperly as sulfur. To save the day, another property of this sulfur excitation is the delay of the emission from when it is initially thermally decomposed. When the combustion process is pulsed, the emitting light will yield the chemiluminescence from hydrocarbons shortly after excitation, whereas sulfur emission is delayed longer. The pulsed flame photometric detector (PFPD) is designed to take advantage of this emission time difference. Figure 1 illustrates a snapshot of the process showing the emissions of the flame ignition, the hydrocarbon chemiluminescence and the desired sulfur emission.

The PFPD offers a number of significant improvements over the conventional flame photometric detector (FPD):

- Increased detectivity
- Increased selectivity (especially relative to hydrocarbons)
- Decreased hydrogen and air consumption

The combustion chamber inside this detector is carefully designed for hydrogen and air to premix and then build up inside the detector to a point that they detonate from the constantly glowing ignitor close by. The resulting ignition propagates down the chamber and self-extinguishes at the bottom. The air/hydrogen mix fills up the chamber again and the process repeats. The setup causes the explosion to occur about every 400 milliseconds and lasts for about 2 millisecond, yielding a regular “popping” noise that is the audible characteristic of this detector. The hydrocarbon chemiluminescence occurs soon after the “pop”, typically from 2 to 6 milliseconds after, and is normally finished by the time sulfur specie starts emitting. A special electronic gate is set to selectively detect only the sulfur emission, typically from 6 to 26 milliseconds after ignition, with the ignition and hydrocarbon responses ignored by a delay in the activation of the gate. A trigger level is set to initiate the signal processing upon ignition of the flame so that the gate window for the sulfur signal is monitored consistently, despite slight changes in the pulse frequency of the flame. The process repeats typically near 4 hertz to generate a chromatographic peak for the target compound.

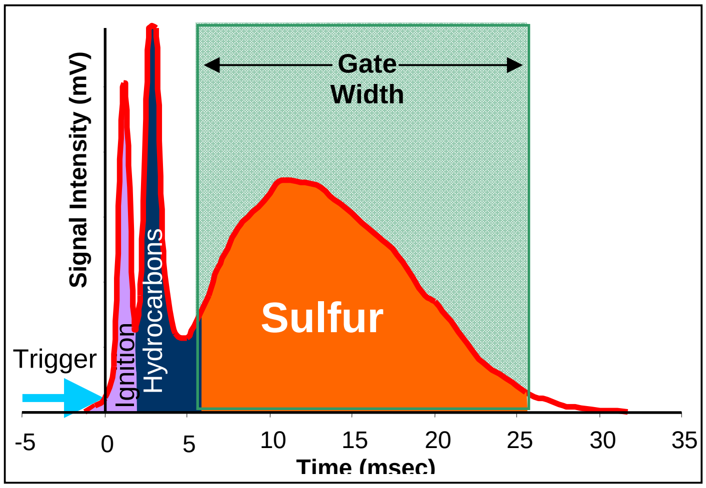


Figure 1. Characteristic pattern of emissions from PFPD.

A split valve is used to finely adjust the air/hydrogen flow rate through the combustor section of the PFPD. “Tick-tock” occurs when the combustible mixture in the ignitor chamber alternates between ignition in the chamber and then the next cycle fails because the combustor has not yet filled up with a combustible gas mixture. Optimum performance is generally achieved when the detector is operating near, but not in, the tick-tock mode. Establishing tick-tock and subsequently the proper setting of the air-hydrogen needle valve is accomplished visually using the scope mode in the PFPD Tuning Application, and adjusting the needle valve from closed (fully clockwise) and increasing flow with the valve knob in a counterclockwise rotation. The display will eventually alternate between seeing the full emission signal, and then the next pulse will have the signal disappear - “tick-tock”. When this is detected, the split valve is advanced one-half turn more counterclockwise to create a steady signal, but create a flame as lean as possible, for maximum response and best selectivity for the target element.

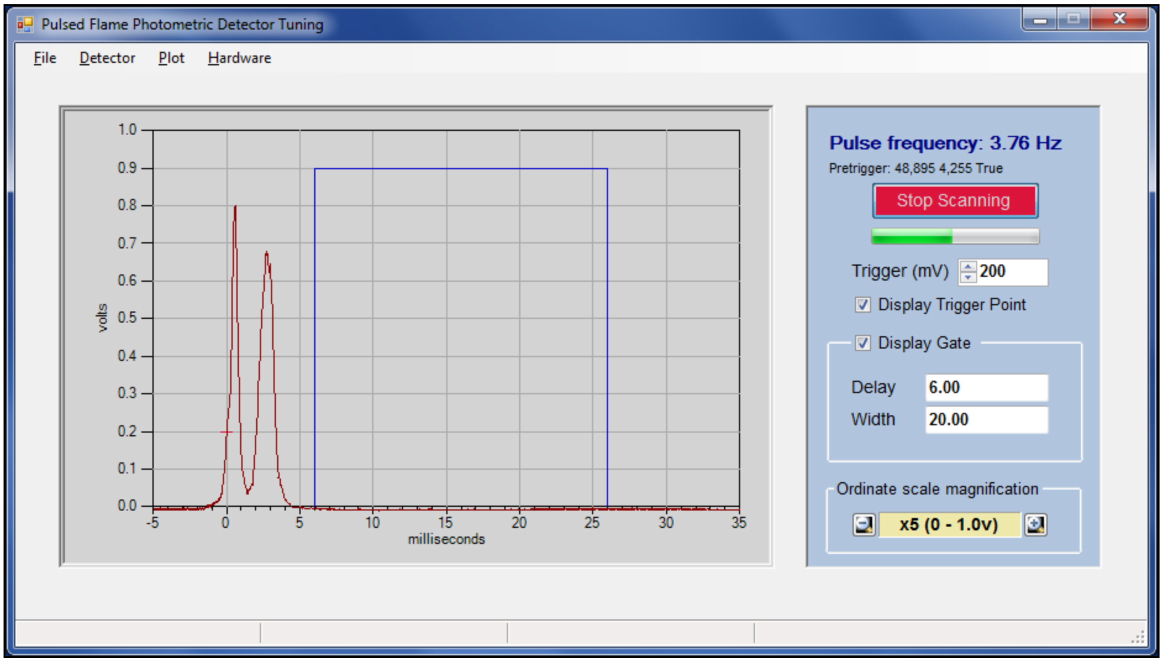
PFPD Tuning Application is also useful in monitoring encroachment of the hydrocarbon emission into the “sulfur” window during the chromatographic process, defined by the gate parameters. A large hydrocarbon peak could be improperly attributed to “sulfur”. The gate delay can be increased to exclude this hydrocarbon.

SYSTEM REQUIREMENTS

- Operating System - Windows 7 or XP.
- Workstation Compatibility - Scion Compass, Varian Galaxie, Scion MS Workstation, Varian Star Workstation.
- Gas Chromatographs - Scion 456/436, Varian/Bruker 450, Bruker 451, Varian 3800.

PACKAGE CONTENTS

- Application CD, with Measurement Computing InstaCal Drivers.
- Measurement Computing USB-1608FS-Plus Data Acquisition Device.
- Cable for attachment to PFPD Electrometer, and USB cable from 1608FS to Computer.



Pulse frequency: 3.76 Hz

Pretrigger: 48,895 4,255 True

Trigger (mV) 200

☑ Display Trigger Point

☑ Display Gate

Delay 6.00

Width 20.00

Lotus Consulting
310/569-0128
email randy@lotusinstruments.com

5781 Campo Walk
Long Beach, California 90803
www.lotusinstruments.com