

The example data presented here represents typical expected performance and is provided for the purposes of illustration and user guidance only. All Waters analytical instruments are installed and tested in accordance with Waters Standard Performance Tests.

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XEVO TQ-XS APGC/MRM

System Sensitivity – Positive Ion APGC

This document presents example data obtained using an APGC - Xevo TQ-XS system when performing a pulsed splitless injection, showing the sensitivity of the instrument. The test is based on monitoring the MRM response for the injection of a solution of TCDD. The details of this test are given below.

EXPERIMENTAL CONDITIONS

Sample TCDD – 1fg/μL in Nonane (1fg on column)

GC Conditions

This analysis was performed using a DB-5MS (30m x 0.25mm x 0.25μm) and a split/splitless injector. The following GC parameters were used:

A 1μl injection was completed using the pulsed splitless mode.

Helium carrier gas flow:	2.0 mL/min.
Injection:	1μl pulsed splitless
Transfer Line:	280 °C
Injector Temperature:	290 °C
Pulse Time	0.5 mins
Pulse Pressure	32 psi
Purge Flow:	30 mL/min
Purge time:	1.2 min
Septum purge:	3 mL/min
Initial Oven Temp:	130 °C
Initial Time:	1.2 min
Rate 1:	20 °C/min
Final Temp 1:	320 °C
Hold time 1:	3.3 min
Total Run time 1:	14 mins

MS Conditions

Instrument:	Xevo TQ-XS
Ionization mode:	APGC positive ion
MRM transition:	321.9 m/z to 258.9 m/z 319.9 m/z to 256.9 m/z
Dwell time:	100ms
Interscan delay:	Auto
Span:	0 Da
Resolution:	Peak width at half height of precursor and product ions <0.7 Da
MRM spike rejection:	not enabled

APGC Ion Source conditions:

Cone gas:	270 L/hr
Aux flow:	200 L/hr
Sampling cone:	35V
Source offset :	30V
Makeup flow on GC:	250 mL/min
Collision energy:	30V

Software

MassLynx 4.2 SCN 943

Signal to noise calculation

Signal is defined as the height of the chromatographic peak of interest (9.53 min) and noise is defined as the Pk-Pk (ignore scan outside +/-2SD, ignore zeros) of a continuous section of the mass chromatogram (8.0-9.0 mins).

RESULTS

For an on-column injection of TCDD (1fg) using a gradient separation, monitoring both the MRM transitions 321.9 m/z to 258.9 m/z and 319.9 m/z to 256.9 m/z in positive ion mode, the chromatographic signal to noise on average was greater than 28:1 for ten acquisitions (See Figure 1) with negligible response in the blank response (bottom chromatographic trace). Figure 2 demonstrates <10% RSD for peak areas for ten consecutive injections.

1fg/μL TCDD

160210 APGC TCDD 025 Sm (Mn, 2x2)

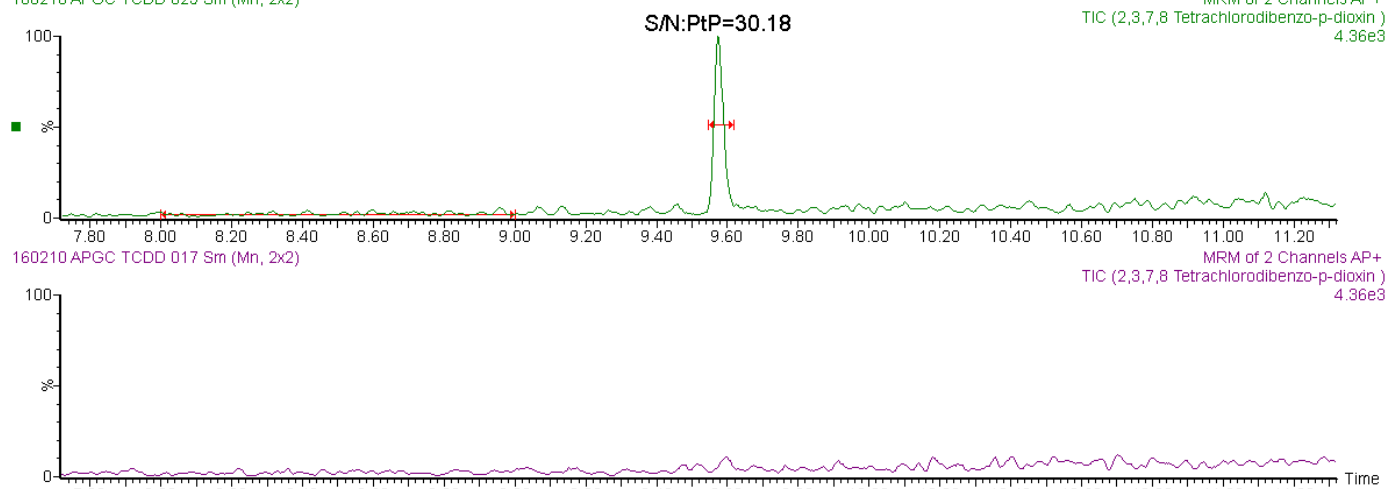


Figure 1: Signal to noise measurement of 1fg TCDD on column, in addition to a solvent blank injection.

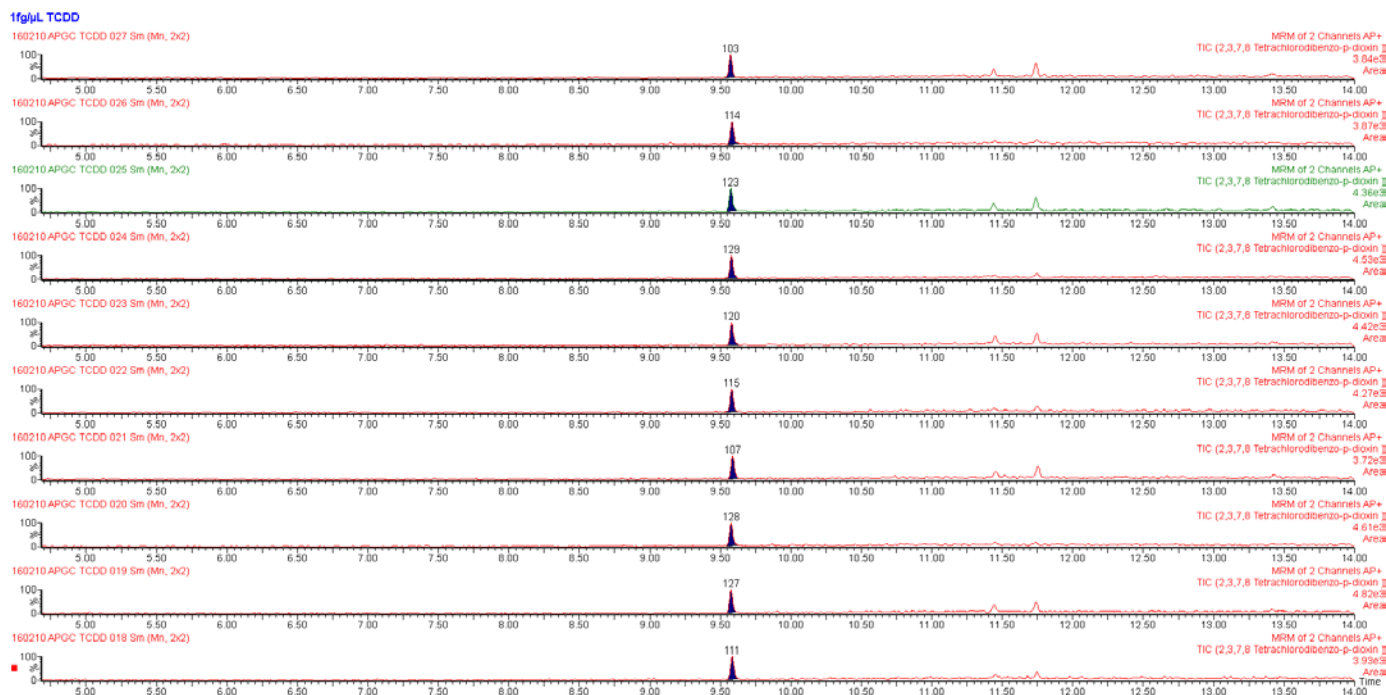


Figure 2: Peak area reproducibility over ten replicate injections of 1fg TCDD on column.

Injection number	Area	Ion Ratio
1	111	4.5
2	127	14.7
3	128	14.9
4	107	11.9
5	115	10.6
6	120	-13.7
7	129	-5.1
8	123	-7.7
9	114	-10.5
10	103	1.4
Mean	119.6	
SD	8.8	
%RSD	7.4	

Table 1: Peak area reproducibility for ten replicate injections of 1fg TCDD on column in addition to the percentage error of the ion transition ratio ($319.9 > 256.9$ to $321.9 > 258.9$ m/z) against the theoretical value of 0.959.

Table 1 shows the %RSD of the 10 injections to be <10%. In addition the ion transition ratio for all ten injections is within 15% of the theoretically determined ion transition ratio.



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