# **Supplementary Information**

# Gas Chromatography-Mass Spectrometric Identification of Cyanide Using a Nucleophilic Substitution Based Derivatization with *s*-Phenyl benzenethiosulfonate

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## **Supplementary Information:**

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#### 1. General details

2. Figures

Reagents were obtained from commercial supplier, and used without further purification. Solvents used were purified by standard methods prior to use. The GC-MS analyses were performed on Agilent 6890 GC, with model 5973 mass selective detector. NMR spectra were recorded on a Bruker AVANCE II 400 MHz. TLC analysis was performed on silica gel 60  $F_{\rm 254}$  plates and column chromatography was conducted over silica gel (mesh 100-200).



# Figure-S1: a) Derivatized product phenyl thiocyanate in organic extraction b) Trimethyl silyl and c) methyl derivatives of reaction product Phenyl sulfinic acid in aqueous layer



Figure-S2: Concentration Vs Relative response of derivatized product





Bottom: Total ion chromatogram of ethyl acetate extraction of 5 ppm cyanide sample of tap water after treating with derivatization reagent. (derivatized product phenyl thiocyanate at 7.39 min)



**Figure-S4:** Top: Total ion chromatogram of ethyl acetate extraction of sample of apple seed extraction after treating with derivatization reagent.

Bottom: Mass spectrum of cyanide derivatized product phenyl thiocyanate at 7.379 min)

#### 3. Derivatization procedure for cyanide from apple seed extraction:

1 gr of apple seeds were crushed using a mortar, and ground for 15 minutes vigorously to get a paste like substance. To this 2 ml of deionized water was added and mixed well. This mixture is allowed to stand at room temperature for 5 hrs in a closed vial. The mixture is centrifuged at 5000 rpm for 20 minutes. The supernant water layer was separated. To the 0.5 mL of this supernant water layer 0.1 ml of derivatization reagent in methanol was added. The resultant turbid solution was shaken well for 5 minutes. 0.25 mL of ethyl acetate was added and agitated for 10 minutes, an emulsion was formed. In order to separate the ethyl acetate layer, the resultant emulsion was centrifuged for 20 minutes at 5000 rpm. The ethylacetate layer (upper) was separated and isolated and analysed using GC-MS. The results confirm the presence hydrogen cyanide by identifying the derivatized product phenyl thiocyanate.



#### 4. Selected copies of <sup>1</sup>H, <sup>13</sup>C NMR spectra

Figure-S5: <sup>1</sup>H NMR spectra of derivatization reagent *s*-phenyl benzenethiosulfonate



Figure-S6: <sup>13</sup>C NMR spectra of derivatization reagent *s*-phenyl benzenethiosulfonate

S.No.	Derivatization reagent	Analytical	Limit of	Limit of	Derivatization	Pre-
		Technique	Detection (LOD)	Quantitation	reaction	concentration
				(LOQ)	conditions	
1.	Pentafluoro benzylbromide		0.5 μg/mL	0.2 μg/mL	38/30min	
	(polymer bound phosphonium	GC-MS	(Full Scan)	(SIM)		
	salt as catalyst)					
	(Ref-24)					
2.	Two step derivatization		0.01 μg/mL	0.02 μg/mL	Rt/20min	Evaporated to
	1.Benzaidehyde	GC-MS	(SIM)	(SIM)		nearly drying
	2.heptafluorobutyryl chloride					
	Ref-26					
3.	1. cysteine, 2. Hypochloric acid		0.07 μg/L	0.2 μg/L	Rt/ 10 min	
	(Ref-27)	LC-MS				
4.	2-(dimethylamino)ethanethiol	GC-MS/MS	0.02 μg/mL	0.07 μg/mL	60/ 20 min	10ml water to
	(Ref-30)					100 EtOAc (100
						times)
5.	s-Phenyl benzenethiosulfonate		0.075 μg/mL	0.25 μg/mL	Rt/ 5min	No pre-
	(present study)	GC-MS	(SIM)	(SIM)		concentration

### Table-S1: Comparison of reported methods for detection of cyanide and preset study