METHOD FOR DETERMINATION OF POLYCHLORINATED DIBENZO-*p*-DIOXINS (DIOXINS)/ POLYCHLORINATED DIBENZO FURANS (FURANS) IN PALM OIL PRODUCTS

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SCOPE

Development of a method for determination of polychlorinated dibenzo-*p*-dioxins (Dioxins)/polychlorinated dibenzo furans (Furans) in palm oil products.

BENEFIT

The developed method can be applied for analysing and monitoring the levels of dioxins/furans in palm oil products.

INTRODUCTION

Polychlorinated dibenzo-*p*-dioxins (PCDDs) and polychlorinated dibenzo furans (PCDFs), collectively known as dioxins are major persistent organic pollutants (POPs) that are found widely all over the world. Centres for Disease Control has classified this chemical compound as toxic, stable, do not break down easily and ubiquitously presence in the environmental and food matrices. *Figure 1* shows the chemical structures of dioxin and furan.

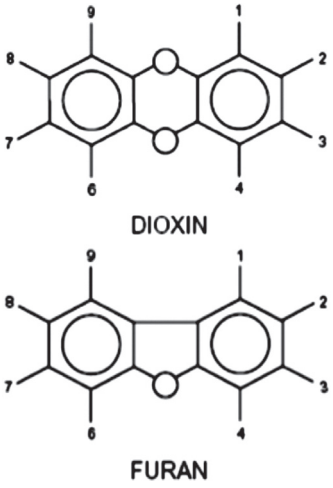

Figure 1. Chemical structures of dioxin and furan.

Dioxins are formed unintentionally as a by-product of industrial waste, chemical processing, incineration process and human activities such as open burning. This toxic and persistent compound then spread into the environment and tend to accumulate in the fatty and organic rich matrices such as foods. Currently, food is one of the major sources of human exposure to dioxins due to the lipophilicity nature of the compound and resistance to metabolic degradation. More than 90% of human exposure to dioxins is derived from foodstuffs. Foods, particularly dairy products, meat, oils/fats and fish have been identified as the primary immediate sources of intake of dioxins for the general population. Dioxins may form in palm oil product during processing (at the milling or refining) or through fall-out from surrounding environment that has high level of dioxin content. The palm oil industry is required to analyse for dioxins in palm oil products in order to comply with international food safety regulation. European Union (EU) has set maximum level of dioxin content as low as below 0.75 WHO TEQ pg g^{-1} for vegetable oil intended for EU countries. Thus, monitoring of dioxin content in palm oil products is pertinent to ensure Malaysian palm oil products are safe for human consumption and that the levels are within the maximum levels imposed by the EU and other importing countries.

METHOD PERFORMANCE

Calibration Curve

Calibration of target congeners was carried out using five calibration solutions namely CS1 to CS5. Each calibration solution has different concentration of the native ^{13}C dioxins/furans congeners which ranges from 0.5 pg μl^{-1} to 200 pg μl^{-1} (Tetra CDD/F); 2.5 pg μl^{-1} to 1000 pg μl^{-1} (Penta-Hexa-Hepta CDDs/Fs) and 5.0 pg μl^{-1} to 2000 pg μl^{-1} (Octa CDD/CDF). Calibration curve was es-

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METHODOLOGY

Sample
(palm oil products)

↓ ← Add

N-hexane
(stir to dissolve)

↓

Acid hydrolysis

↓ ← Transfer

Clear supernatant into pear shape flask

↓

Concentrate using rotarvapor

↓ ← Add

Distilled water

↓

Organic phase (analyte)/water

↓ ← Pipette out water

Organic phase (analyte)

↓

Power Prep™ Clean up System (*Figure 2*)

↓ ← Rotarvarp

Transfer into tapered vial

↓

Concentrate using dry block under nitrogen blow

↓

GC-HRMS Analysis
(*Figure 3*)

tablished through several injections of the calibration solution (CS1-CS5). The linearity of the target congeners was good with $0.999856 \leq R^2 \leq 0.999998$. *Figure 4* shows the chromatogram of the calibration solution (CS) 4 and *Figure 5* shows calibration curve for 2,3,7,8-TetraCDD congener.

Figure 2. Power Prep™ Clean up system supplied by Fluid Management System.

Figure 3. Gas chromatography – high resolution mass spectrometry (GC-HRMS) supplied by Thermo Fisher Scientific.

Limit of Detection (LOD) and Limit of Quantification (LOQ)

Limit of Detection (LOD) for developed method was established based on 3:1 signal to noise ratio while Limit of Quantification (LOQ) was based on 10:1 signal to noise ratio. LOD and LOQ for dioxins/furans were 0.07 WHO-TEQ pg g^{-1} and 0.37 WHO-TEQ pg g^{-1}, respectively.

Percentage Recovery

The recovery of the congeners from oil matrix analysis was carried out at concentration of 1.25 pg g^{-1}. *Table 1* shows the recoveries for dioxins/furans congeners. Percentage recoveries for all congeners were in the range 60%-120% and this falls within the EU Regulation No. 252/2012 requirement.

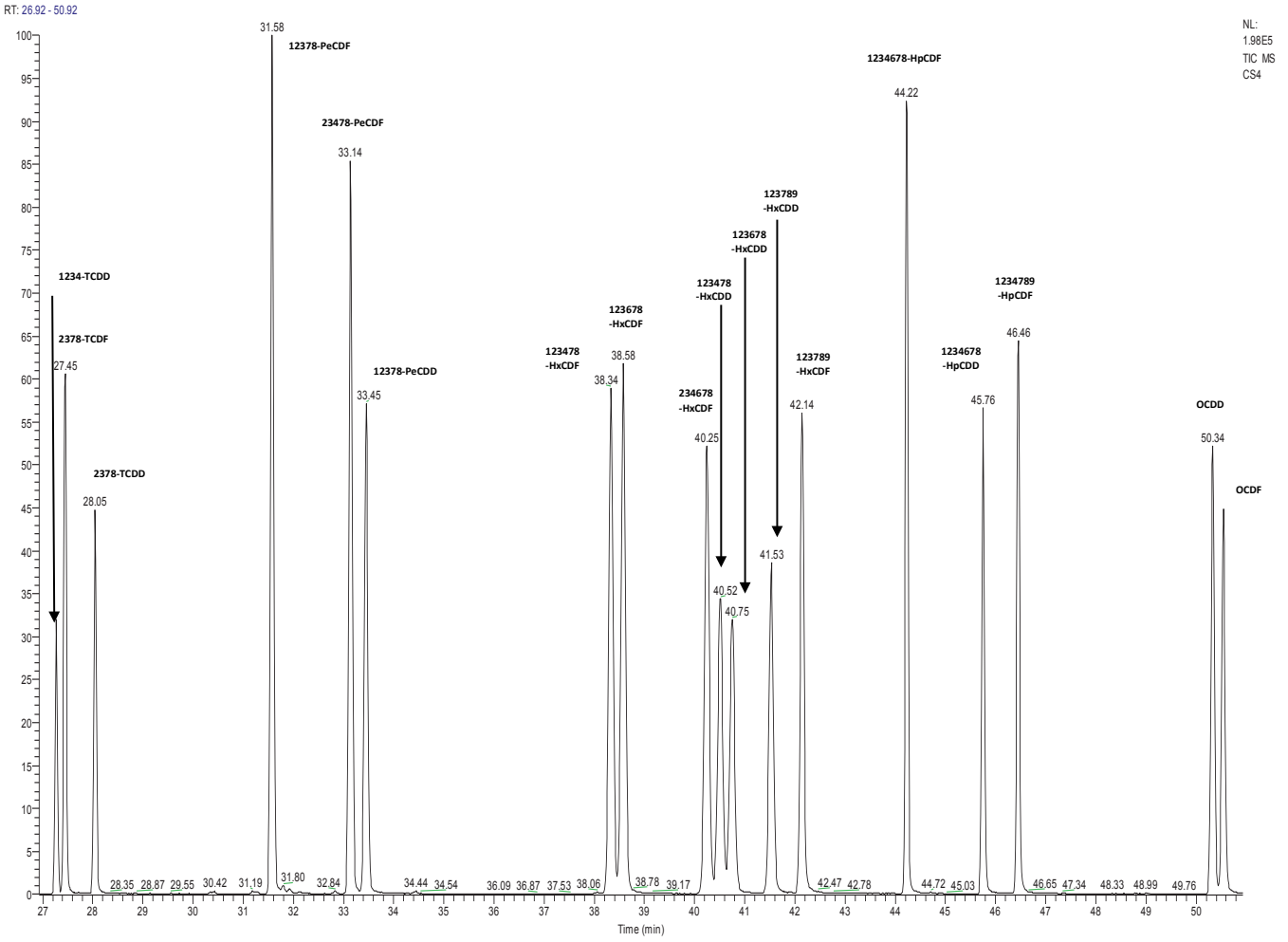

Figure 4. Chromatogram of the calibration solution (CS) 4.

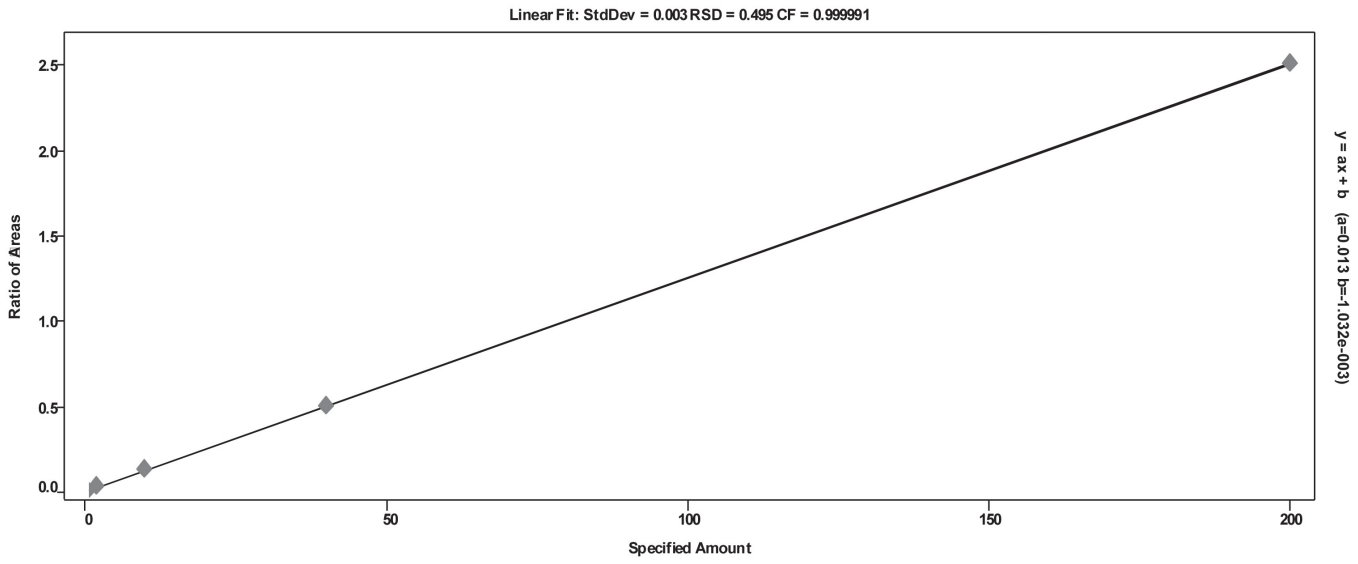

Figure 5. Calibration curve for 2, 3, 7, 8 - Tetra CDD congeners.

TABLE 1. RECOVERIES FOR DIOXINS/FURANS CONGENERS

Congeners	1.25 pg g^{-1}
2,3,7,8–TCDD	66
2,3,7,8–TCDF	105
1,2,3,7,8–PeCDD	62
1,2,3,7,8–PeCDF	65
2,3,4,7,8–PeCDF	65
1,2,3,4,7,8–HxCDD	64
1,2,3,6,7,8–HxCDD	68
1,2,3,4,7,8–HxCDF	103
1,2,3,6,7,8–HxCDF	92
1,2,3,7,8,9–HxCDF	87
2,3,4,6,7,8–HxCDF	88
1,2,3,4,6,7,8–HpCDD	84
1,2,3,4,6,7,8–HpCDF	101
1,2,3,4,7,8,9–HpCDF	90
OctaCDD	61

Repeatability

Repeatability study was carried out by extracting and analysing five replicate of RBD palm olein samples to evaluate the accuracy of the method. Mean concentration of RBDo analysed was 0.17 WHO-TEQ pg g^{-1} with RSD of 8.3%, which is below 10%.

INDICATIVE COST

The cost of analysis per sample is RM 2000 and is subject to change.

For more information, kindly contact:

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