

SCOPE

This test method describes a quantitative analysis method for determination of Indaziflam residue in palm oil products using High Performance Liquid Chromatography (HPLC) and Mass Spectrometry Detection (LC-MS/MS).

INTRODUCTION

Indaziflam is a herbicide belonging to chemical class of alkyltriazines. The advantage of herbicide is its low application rate due to long lasting actions and coverage of broad spectrum weeds such as bluegrass, goose grass, ryegrass and goosefoot species, which are difficult to eliminate (Pesticide Fact Sheet, 2010). It is registered as herbicide in some countries and in the process of being registered on a global scale (Pesticide Fact Sheet, 2010; Public Release Summary, 2015; Department of Agriculture). Therefore, Indaziflam is used in agriculture for the management of weeds. However, high doses or frequent use of Indaziflam may lead to contamination of crop and thus affect yield. Therefore, in order to minimise the consumer's exposure to this pesticide through

food contact, the monitoring of this chemical is of importance.

Analysis of Indaziflam and its metabolites includes a Liquid Chromatography (LC) method with Ultraviolet (UV) (Revathi *et al.*, 2017) or Mass Spectrometry (MS) detection methods (Hu *et al.*, 2018), as reported in some literature on food matrices. Revathi *et al.* (2017) reported a method for the detection of Indaziflam and its metabolite (diamino triazine) in the black grape matrix by HPLC-UV, which requires an excessive amount of sample and solvent, lengthy extraction as well as analysis time. In 2018, Hu *et al.* published a method for soil, water and fruits using modified QuEChERS, that requires clean up by octadecylsilane and HLB cartridges. Recently, Liu *et al.* (2019) published a method in pitaya matrix that only requires 24 min for HPLC detection. However, this method is typically laborious and cost-intensive.

Thus, a fast and robust analytical method for targeting simultaneous determination of Indaziflam and its metabolites has been established in MPOB for series of palm products *i.e.* palm oil, crude palm kernel oil, crude palm oil, RBD palm oil, and RBD palm olein.

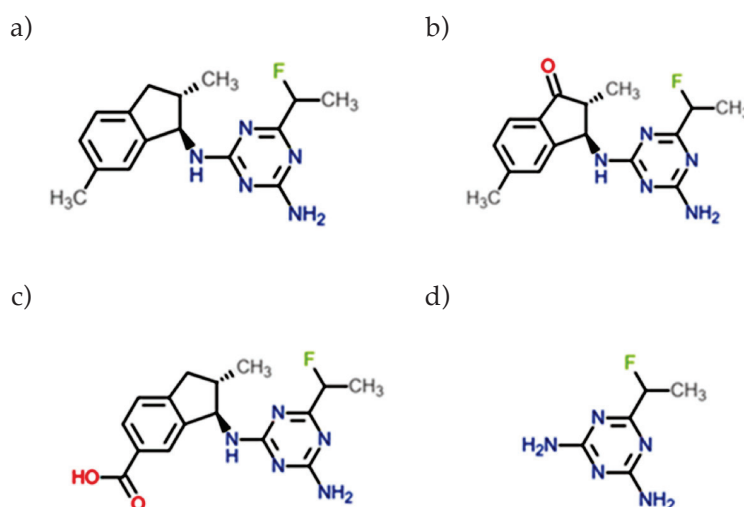


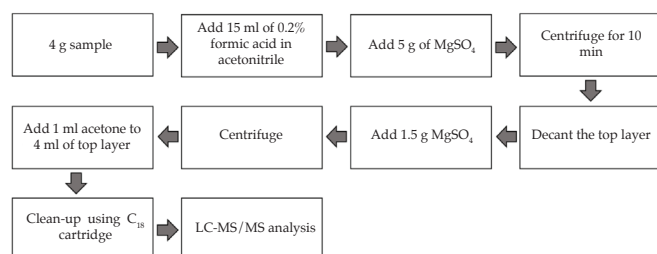
Figure 1. Chemical structure of Indaziflam (a), IND-Triazine indanone (b), IND-Carboxylic acid (c) and IND-Diaminotriazine (d).

PRINCIPLE

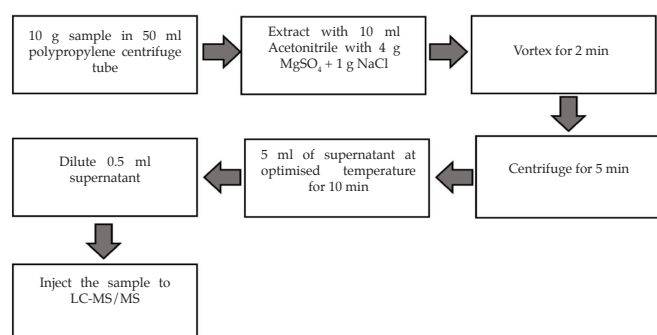
Analysis of Indaziflam and its metabolites in palm oil and palm oil products can be performed within a chromatographic run time of 7 min using liquid chromatography tandem mass spectrometry (LC-MS/MS). The optimised sample preparation workflows are (a) QuEChERS with clean up at -80°C , and (b) Acetonitrile based method- clean up using C_{18} cartridge; both methods provide satisfactory performance. The optimised chromatographic method, *i.e.* acidification of mobile phase and change in gradient programmed provides better retention, sufficient selectivity and good sensitivity for all the test analytes, and minimises the matrix effects. The method performance is of satisfactorily at 0.01 mg kg^{-1} , where higher levels in compliance with the SANTE/11813/2017 guidelines of analytical quality control. The developed method is potential for analysing Indaziflam and its metabolites in a single chromatographic run. As the method demonstrates good selectivity, sensitivity, robustness, accuracy, and preciseness, it can be widely implemented for routine residue testing laboratories.

METHODOLOGY

Acetonitrile-based Method



QuEChERS Method



RESULT

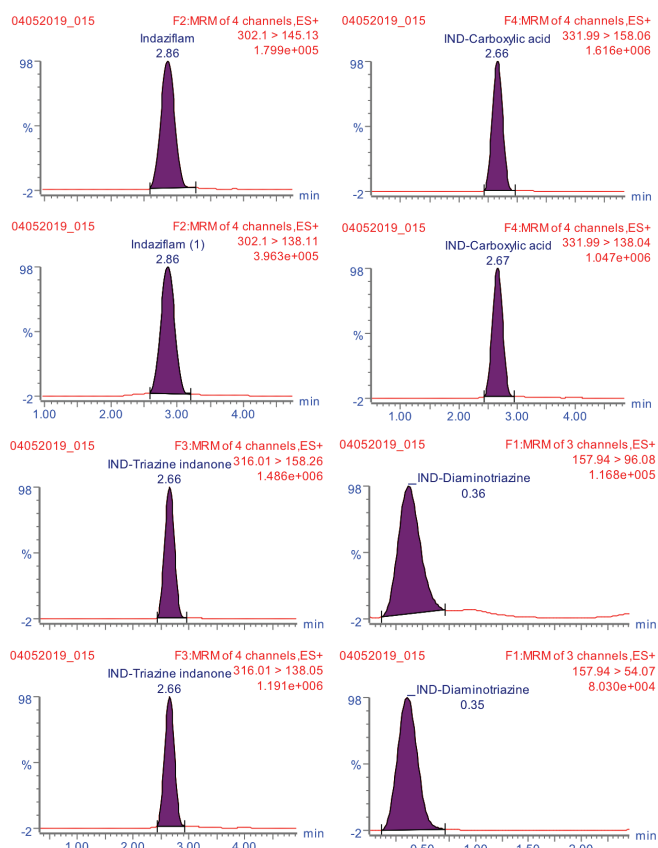


Figure 2. Chromatograms of spiked CPKO sample, at LOQ level (for Indaziflam, IND-carboxylic acid, and IND-triazine indanone, the LOQ was 0.001 mg kg^{-1} , and for Diaminotriazine, the LOQ was 0.01 mg kg^{-1}).

SERVICES OFFERED

- Method of analysis to be transferred to palm oil industry.
- To receive palm oil samples for analysis of Indaziflam residue.

ADVANTAGES

- Reliable and sensitive method of analysis;
- Analysis of 24 samples per day;
- Less solvent and time consuming; and
- 'Green' method of analysis.

CONCLUSION

The method can be easily applied for routine analysis to determine Indaziflam and its metabolites in palm oil product. The LC-MS/MS instrument used for the analysis can be easily maintained and optimised to meet the required conditions.

TABLE 1. RESULTS FOR RECOVERY STUDY FROM SPIKED SAMPLES AT KNOWN CONCENTRATION LEVEL

Compound	Indaziflam		Diamino triazine		Triazine indanone		Carboxylic acid	
	0.001 mg kg ⁻¹	RSD (%)	0.001 mg kg ⁻¹	RSD (%)	0.001 mg kg ⁻¹	RSD (%)	0.001 mg kg ⁻¹	RSD (%)
RBD palm olein	95.4	5.2	112.4	2	118.6	10.1	112.8	7.1
RBD palm oil	105.9	5.5	95.3	5	104.1	6.8	107.8	7.3
Crude palm oil	88.5	9.3	86.6	4	93.1	8.4	75.4	2.5
Crude palm kernel oil	81.6	2.6	114.8	4	107.7	12.3	82.6	5.6

INDICATIVE COST

The cost of analysis is RM500 per sample, including sample preparation and analysis. The cost is subjected to change without prior notice.

REFERENCES

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