

## SCOPE

This test method describes a quantitative method of analysis for determination of paraquat residue in oil matrix by High Performance Liquid Chromatography (HPLC) and UV detection.

## INTRODUCTION

Paraquat is the common name for 1,1-dimethyl-4,4-bipyridilium dichloride,  $C_{12}H_{14}Cl_2$ . The analysis of paraquat in food crops using the ion-exchange method has showed that the measurement of light absorption of reduced solutions of paraquat after concentration and purification by cation-exchange chromatography is not practical and less sensitive for detection of paraquat in the oil matrix. Furthermore, the method of extraction and clean-up steps is laborious and time consuming. In-addition, the correction factor of optical density applied to the final calculation of paraquat content contributes to low accuracy and less sensitivity of the method due to degree of linearity of the background adsorption. (Calderbank *et al.*, 1965). Furthermore, the analysis of paraquat using UV-VIS technique is less sensitive and low selectivity. Therefore, a reliable and sensitive method was developed and validated using advanced analytical techniques with high sensitivity and selectivity for determining traces of paraquat in oil matrix.

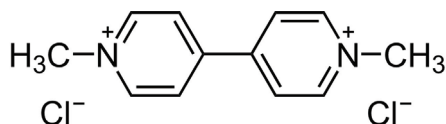


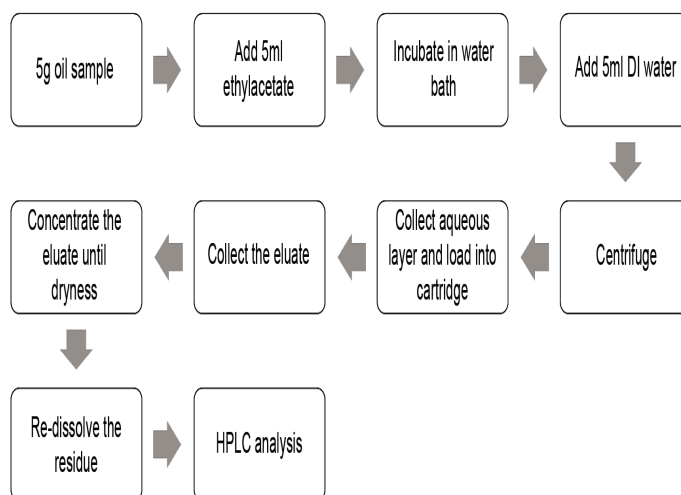
Figure 1. Chemical structure of paraquat.

## PRINCIPLE

Analysis of paraquat residue in oil matrix is based on liquid-liquid extraction and HPLC-UV detection. A new method was developed from

modification of water samples analysis. The modification involved diluting of contaminated palm oil with ethyl acetate and deionised (DI) water. The aqueous layer that contained paraquat residue was subjected to clean-up step with anion-exchange cartridge and concentrated prior to HPLC-UV analysis.

## METHODOLOGY



## RESULTS

Results showed a good linear calibration curve for six concentration levels ranging from 0.025 - 1.0  $\mu\text{gml}^{-1}$  with  $R^2 = 0.9930$ . The limit of detection (LOD) and limit of quantification (LOQ) were 0.025  $\mu\text{gml}^{-1}$  and 0.05  $\mu\text{gml}^{-1}$  respectively. The percentage recovery of paraquat residues from spiked sample at three concentration levels: 0.1  $\mu\text{gml}^{-1}$ , 0.05  $\mu\text{gml}^{-1}$  and 0.5  $\mu\text{gml}^{-1}$  were above 85%.

## SERVICES OFFERED

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

TABLE 1. AVERAGE PERCENT RECOVERY OF PARAQUAT RESIDUE FROM SPIKED SAMPLES (n=7)

Concentration ( $\mu\text{gml}^{-1}$ )/Day	0.05	0.1	0.2	0.4	0.5
1	91	109	79	77	86
2	90	98	72	82	91
3	94	101	91	75	85

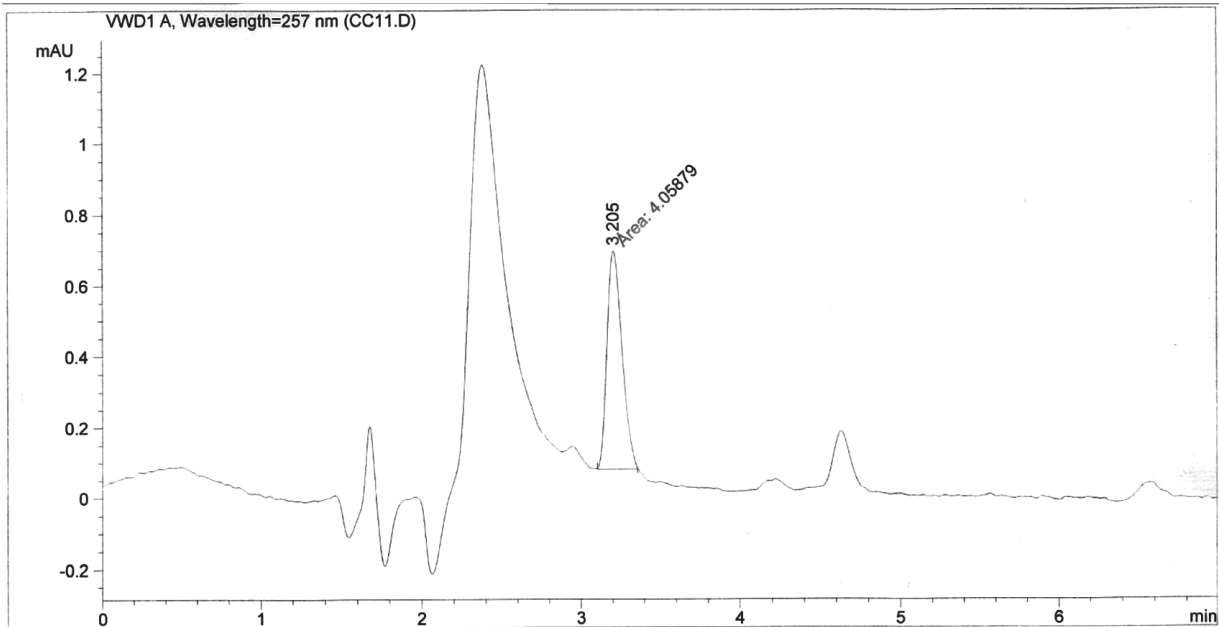


Figure 2. HPLC chromatogram for  $0.025 \mu\text{gml}^{-1}$  paraquat standard solution.

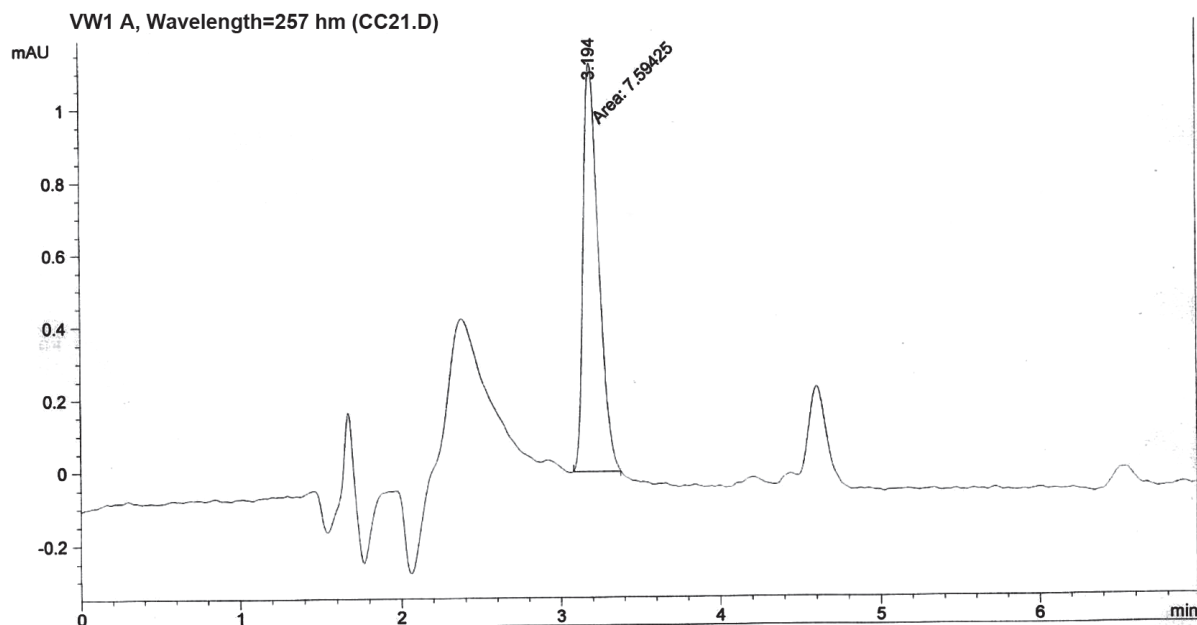


Figure 3. HPLC chromatogram for  $0.05 \mu\text{gml}^{-1}$  paraquat standard solution.

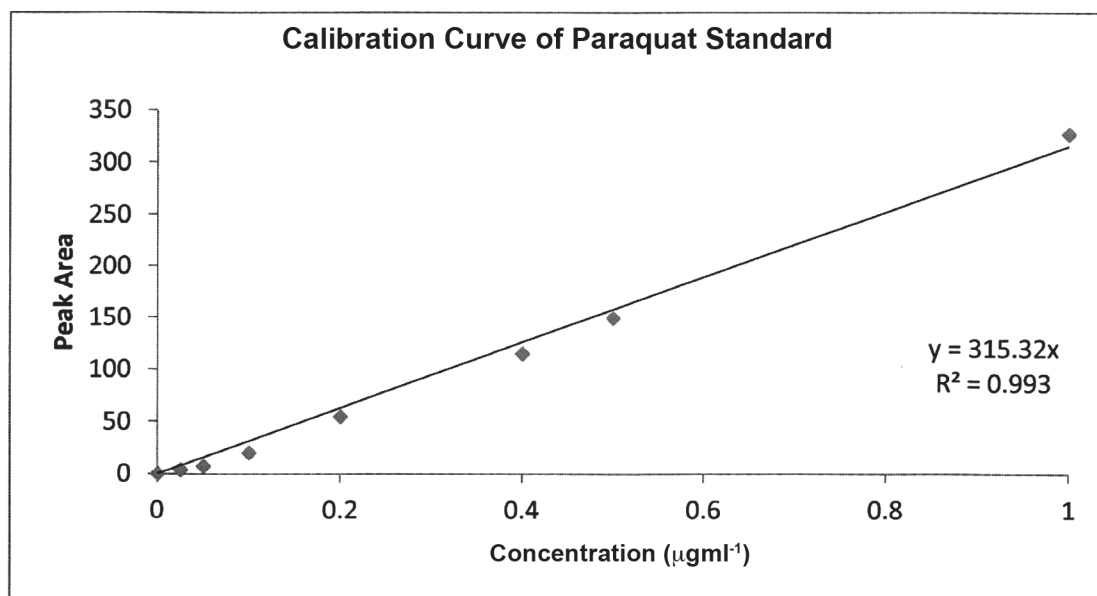


Figure 4. Linear standard calibration curve ranging from 0.025 µgml<sup>-1</sup> to 1.0 µgml<sup>-1</sup>.

### ADVANTAGES

- Less solvent used for extraction of paraquat residue from oil matrix.
- Reliable and sensitive method of analysis.
- 6 samples/day compared to previous method of analysis which is 3 days/sample.
- Less time consuming.
- “Green” method of analysis.

### BENEFITS

- To facilitate palm oil trade to importing countries by meeting CODEX MRL.
- To address food safety issues related to paraquat residue in palm oil.
- To protect consumer’s health.

### CONCLUSION

The method can be easily applied for routine analysis for determination of paraquat residue in palm oil product. The HPLC instrument used for the analysis can be easily maintained and optimised to meet the required conditions.

### INDICATIVE COST

The cost for analysis is RM 350\* per sample, including sample preparation and analysis.  
\* subject to change without prior notice.

### REFERENCES

- CALDERBANK, A and YUEN, S H (1965). An ion exchange method for determining paraquat residues in food crops. *Analyst*, 90: 99-106.
- LORNA, G; BICK, N and PAUL, Y (2002). Liquid chromatography ionization isotope dilution mass spectrometry analysis of paraquat and diquat using conventional multilayer solid-phase extraction cartridges. *J. Chromatography A*, 958: 25-33.
- CASTRO, R; MOYANO, E and GALCERAN, M T (2001). Determination of quaternary ammonium pesticides by liquid chromatography-electrospray tandem mass spectrometry. *J. Chromatography A*, 914: 111-121.

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