

EXTRACTION OF ALKANES FROM PLANT MATERIAL BY SUPERCRITICAL CO₂

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Present methods for extracting and purifying n-alkanes present in the cuticular wax of plants are slow and have a high labour requirement (Mayes *et al.* 1986). A consequence of this is a high analytical cost for experiments using this technology. In the petrochemical, plastics and pesticide industries a new technique for extracting analytes using supercritical CO₂ has been developed. The technique of Supercritical Fluid Extraction (SFE) has the advantages of (1) greatly reducing solvent use (1-5 ml trapping solution), (2) shorter extraction time (3) eliminating the need for further purification and (4) automation. This study sought to determine whether SFE could be used to extract plant alkanes quantitatively and with sufficient purity to allow chromatography without further purification.

SFE extractions were made using a Supelco SFE-400 instrument fitted with a wide-bore restrictor maintained at 120°C. Samples of air dry lucerne chaff (1 g) were hammer milled and alkanes extracted using a range of conditions ranging from 8.2 - 34.5 MPa, and 60 - 180°C for 10 - 30 minutes using either static or dynamic extraction. Extracts were trapped in 5 mL of heptane which was evaporated to less than 2 ml then analysed directly by gas liquid chromatography without further purification. A representative chromatogram is shown in Figure 1.

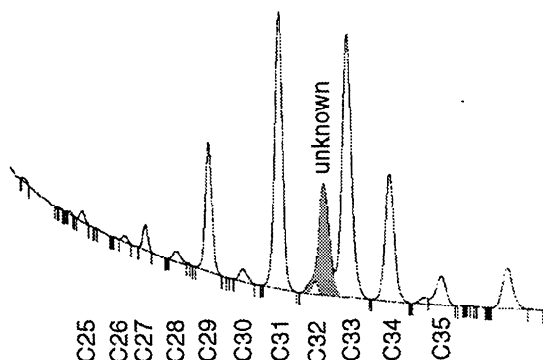


Figure 1. Chromatogram of alkanes extracted from lucerne by supercritical CO₂ at 10.3 MPa and 120°C for 15 minutes using a Supelco SFE -400. The elution peak for C32 from an extract prepared by the method of Mayes *et al.* (1986) is superimposed beneath the unknown peak present in the SFE extract.

Recovery of plant alkanes was incomplete using SFE under all conditions studied. Sample clarity and alkane recovery (mean 73%) were greatest with dynamic extraction at 10.3 MPa and 120°C for 15 minutes. The efficiency of trapping alkanes in heptane as supercritical CO₂ reverted to the gas phase in the vent tube was considered the major source of variability in alkane recovery. Recovery rates were substantially reduced in static compared to dynamic extractions. The levels of background noise from non-alkane analytes were generally low but in SFE extracts a large peak eluted at the same retention time as does the C32 alkane. This peak is not present in conventional solvent extracts of plant alkanes and requires characterisation. SFE offers the opportunity for rapid automated extraction of plant alkanes but further work is required to optimise extraction and minimise contaminants. Thanks to Sigma Aldrich for loan of the test instrument.

MAYES, R.W., LAMB, C.S. and COLGROVE, P.M. (1986). *J. Agric. Sci., Camb.* 107: 161-70.