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Preface

In the mid 1980's supercritical fluid chromatography (SFC) arose with considerable fanfare as the new technique which would shortly "displace HPLC" and become the "universal separation method". After the initial sales and marketing hype had passed and the novelty bandwagon had moved on to capillary electrophoresis, there has still remained a significant group of applications in both chromatography and extraction for which supercritical fluids are the most suitable solvents. These form the basis of the present volume which illustrates by a collection of review and research papers the present state of analytical supercritical technology.

The overall emphasis is on extraction methods, usually as a sample preparation method prior to HPLC or GC analysis, but in some cases the extract is examined directly as the analytical step. This bias on extraction methods reflects the impact of supercritical fluids in the working laboratory and the feeling that in general the sample preparation stages have often limited the through-put of analytical methods. Hence it is an area where advances can have the maximum benefit for the user. Supercritical fluids have been able to offer real advantages over often slow and largely manual methods, in ease of handling and the elimination of organic solvents. Unfortunately the literature has been dominated by applications and few investigators have attempted to study the complex physicochemical factors which control the supercritical fluid extraction (SFE) of real samples

The papers on chromatography are more diverse but also more adventurous, with developments in mobile phases, equipment and detectors suggesting that the instrumentation and technology of SFC has still not reached a satisfactory stage for widespread

adoption. The applications really cover only two areas, of chiral chromatography and metal complex analysis, where SFC offers particular advantages over existing methods.

Overall this collection of papers supports a calmer and more rational view of the state of application of supercritical fluids as their position in the analytical laboratory becoming clearer. Although it was initially claimed that they filled a gap between gases and liquids and "had the properties of both", this was never true. The reality was that they could have the properties of either according to the temperature and pressure but never of both simultaneously. Thus rather than displacing the earlier methods, supercritical fluids have served to link them, providing a continuum of mobile phase properties to the analyst. The blurring of these boundaries is clear in many of the publications. The use of superheated/subcritical water for extraction and chromatography, the role of enhanced fluidity solvents and of pressurised fluid extractions have gone a long way to bridge the gaps in both temperatures and polarity. These papers emphasise that future chromatographers should not be compartmentalised into "gas chromatographers" or "liquid chromatographers" but should view the field as a series of merging separation techniques. Similarly there was a purist view that the fluid had to be "supercritical" to be of value, whereas in chiral chromatography and many other areas, frequently the applications now prefer subcritical conditions.

It has often been the technology of supercritical fluids that have limited this field, the need to handle compressed gases resulted in a need for new equipment. Robust systems only really arrived after the bulk of initial excitement and it was partially the failure of the instrumentation to deliver the repro-

ducibility and reliability that limited the interest in SFC. On the other hand, in these days of mechanical sample handling and high throughput, a lack of reliable automated systems limited the expansion of SFE. But we should not be surprised; HPLC equipment is still developing after nearly 25 years, and supercritical fluid systems have so far only had 5 years to reach a comparable level. For SFE, a greater limiting factor is the lack of education in the theory and practice of sample preparation. Just as analytical chemistry has often been considered an inferior cousin to the “real” fields of chemistry (organic, inorganic and physical), sample preparation is generally regarded as a less valid field of research than

chromatography. The addition of proper coursework in our universities could help to correct this bias.

Rather than be a separate branch of chromatography or extractions it is clear that the use of solvents above their normal conditions of temperature and pressure, up to and including the supercritical state, will continue to expand the range of analytical methods and should be seen as part of an overall spectrum of solubility, polarity and volatility properties of solvents and mobile phases.

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