New Solvents for Extraction

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Using portable extraction equipment in the Bulgarian rose fields

The properties of a new generation of fluorocarbon solvents have been applied to the extraction of plant materials. The core of the solvent is 1,1,1,2-tetrafluoroethane, better known to non-chemists as hydrofluorocarbon-134a or HFC-134a. This product was developed as a replacement for the chlorofluorocarbons that created the aerosal industry and were essential to the spread of low-cost and portable refrigeration. When these propellents and refrigerants were banned because of their ozone-depleting effects, the hunt for replacements resulted in the development of HFC-134a as the best available product. It is now produced by several major chemical companies throughout the world.

Advanced Phytonics Limited, a Manchester, England-based company, has recently filed international patents collectively known as "the Phytonics process" protecting novel procedures using solvents based on HFC-134a. Special blends of HFC-134a and modifying solvents are being produced by Imperial Chemical Industries in Cheshire, England, to meet the particular requirements of the Phytonics process.

In Britain, the UK Ministry of Agriculture recently approved the Phytonics process for the production of natural food flavor extracts. Approval from the Scientific Committee for Foods of the European Union is expected soon.

The Solvents

The boiling point of 1,1,1,2- tetrafluoroethane is -25°C. It is not flammable or toxic. It is not an ozone depleter. It has a vapor pressure of 5.6 bar at ambient temperatures.

By most standards, this is a very poor solvent. For example, it does not mix with mineral oils, triglycerides and (most importantly for our purpose) it does not dissolve plant waxes.

The Phytonics process, therefore, does not produce traditional "concretes" which thereafter must be refined with ethanol to "absolutes." Instead, the products, which we call phytols, are fragrant crystal-clear mobile oils which can be used directly without further physical or chemical treatment.

The process can be used with a range of customized solvents, which we call phytosols. For example, one of the phytosols is exceedingly selective and extracts only highly fragrant, light, clear, mobile oils. Others extract a broader spectrum of components. Where rich, dark, viscous oleoresins or extracts containing triglycerides are demanded, yet another of the new phytosols can be used.

At Advanced Phytonics Limited, we now are modifying the blends by adding small amounts of co-solvents to adapt their dissolving powers to other plant materials such as oleoresins, antibiotics and phyto-pharmaceuticals.

The oils and biological extracts made by this process have extremely low residual solvent residue contents. The residuals are invariably less than 20 parts per billion and are frequently below the levels which can be detected.

These solvents are neither acidic nor alkaline and therefore have only minimal potential reaction effects on the botanical material.

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Figure 1. Capillary gas liquid chromatographic analyses of extracts from *Rosa damascena trigintipetala* (the Kazanlak rose) from the fields of Bulgarsca Rosa Sevtopolis Ltd. at Kazanlak, Bulgaria

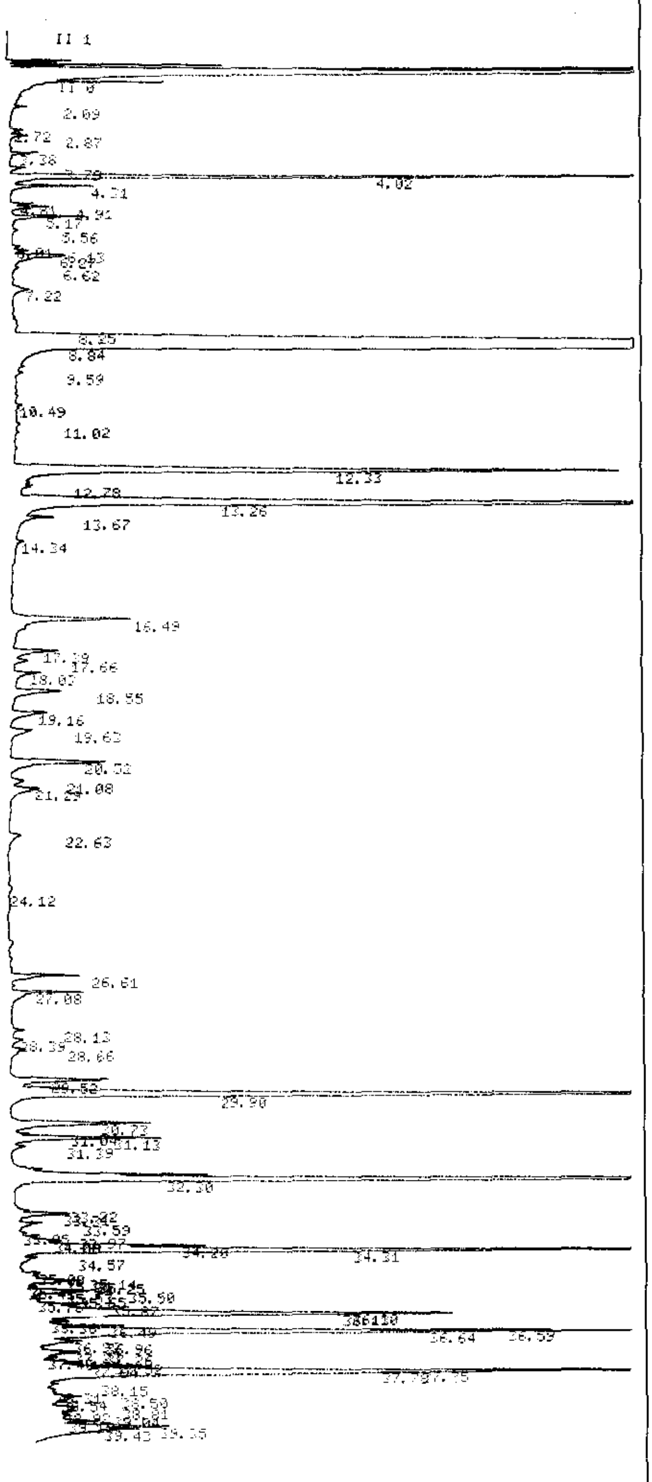

Figure 1.1. A Rose Phytol extracted by the author in the fields using miniature hand-held Phytonics process extraction equipment. The solvent used in this case was Phytosol B

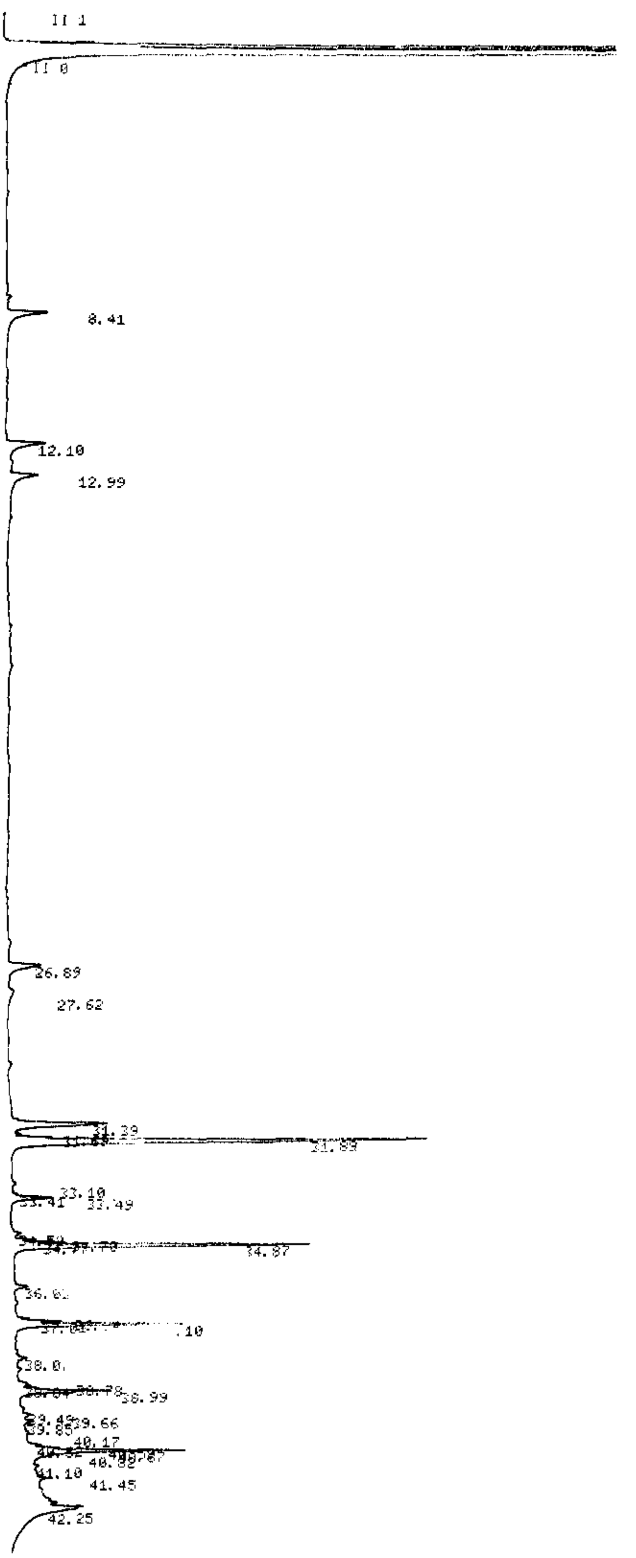

Figure 1.2. The rose concrete routinely produced by Bulgarsca Rosa Sevtopolis Ltd. using the traditional technique of hexane extraction followed by removal of the hexane by distillation at 70°C and 600 mm Hg vacuum

Figure 2. Capillary gas liquid chromatographic analyses of extracts from the rose fields of Bulgarsca Rosa Plovdiv Ltd. at Zelenikovo, Bulgaria

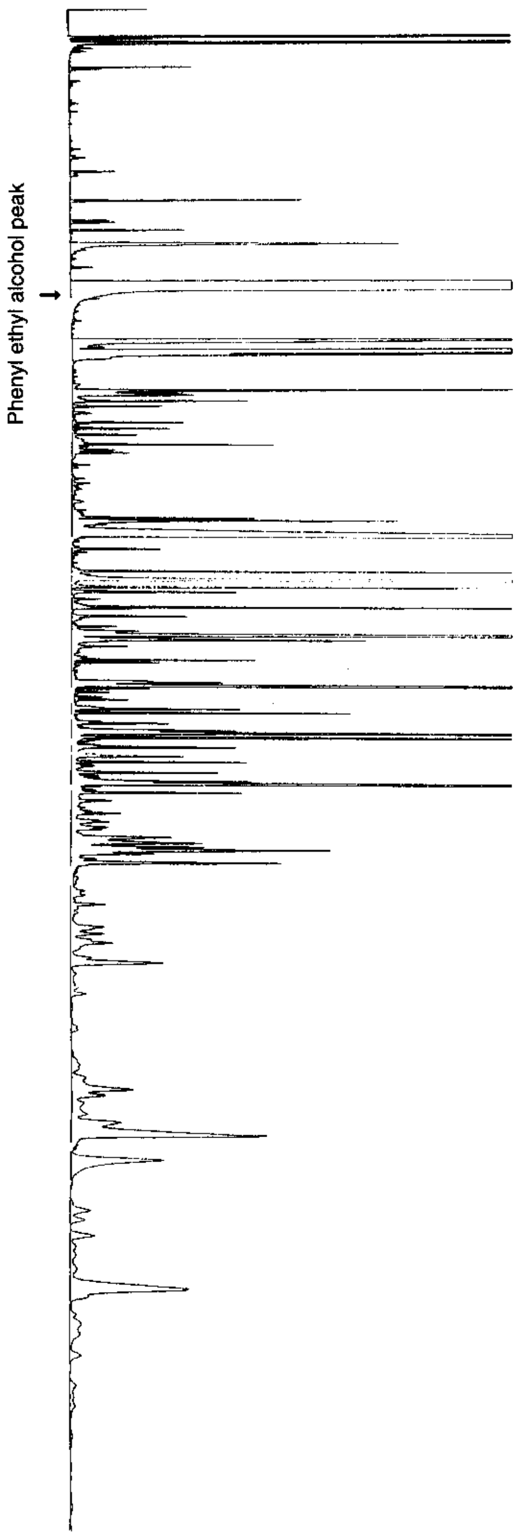

Figure 2.1. A Rose Phytol extracted by the author in the fields using miniature hand-held Phytonics process extraction equipment. The solvent used in this case was Phytosol D

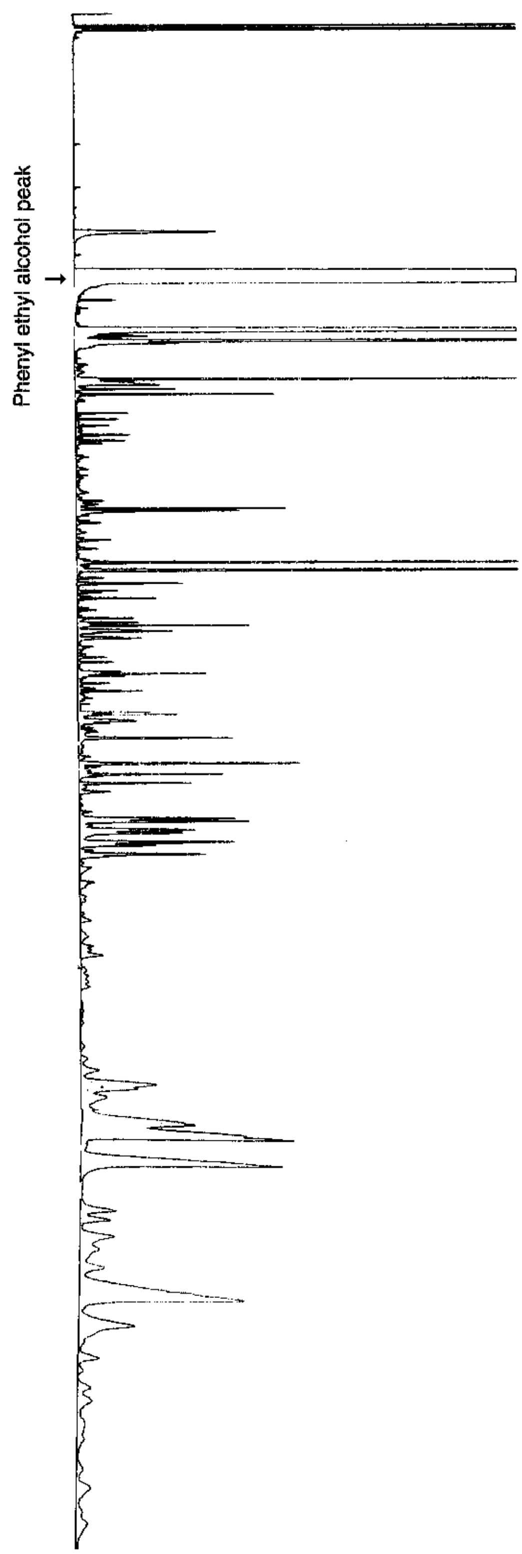

Figure 2.2. The rose absolute routinely prepared by Bulgarsca Rosa Plovdiv Ltd. by trituration, with ethanol, of rose concrete, followed by filtration at sub-zero temperature and then removal of the ethanol by high vacuum distillation

The processing plant is totally sealed so that the solvents are continually recycled and fully recovered at the end of each production cycle. The only utility needed to operate these systems is electricity and not very much of that. For instance, the present portable, skid-mounted 400-liter processing plants operate from a simple domestic (single phase) wall-plug socket and can be operated in primitive environments from a small portable generator.

Even if some of the solvents were to escape, they contain no chlorine in their molecular make-up and, therefore, pose no threat to the ozone layer. Furthermore, they are not VOCs and have a zero potential for generating photochemical ozone.

The waste bio-mass from these plants is dry and pleasant to handle. It is in demand from farmers who use it as a beneficial mulch for enriching their land, as it contains all of the nutrients, other than oils, that went into the process.

To date, the Phytonics process has been used to extract more than 200 crops, including fresh flowers, bark roots, leaves and seeds. Additionally, marine organisms, algae, lichen, molds and fungal cultures, yeasts and bacterial cell cultures are amongst other raw materials which have been extracted to yield antibiotics and other phyto-pharmacologically active products.

The Process

The Phytonics process is simple to understand and operate. The equipment is robust, reliable and easy to maintain. Being a low pressure process, it represents little hazard to semi-skilled operators.

We have built a small handheld extraction unit that requires no external power supply. Since the process is pressure-driven, an extraction run can be operated off a pressure tank of solvent as long as the pressure remains. In a small unit, only a very small amount of extract will be realized, normally less than one gram, but enough to smell and to analyze. After the extraction, the solvent is allowed to evaporate into the air and the extract remains.

Recently, I took such a unit into two rose fields in Bulgaria to produce a small sample of the rose extract from rose blossoms in the two fields immediately after the roses were picked. In each case the solvent was allowed to evaporate. Then the extract was analyzed and the results were compared with the analyses of two commercial oils—a Bulgarian rose concrete and a Bulgarian rose absolute—from the same fields.

The analyses in Figure 1 show that the rose extract, which we call a Rose Phytol, from the Phytonics extraction contained 94 components while the regular rose concrete from the same field contained 34.

In the analyses of Figure 2, note that few peaks can be observed in the traditional rose absolute prior to the emergence of the phenyl ethyl alcohol peak. This should be compared to the abundance of highly volatile components to be seen in the Rose Phytol sample. This is despite the fact that the injected sample of traditional absolute was larger than that of the Rose Phytol, as can be seen by comparing the area under the phenyl ethyl alcohol peak.

Summary

New solvents based on Hydrofluorocarbon-134a and a new technology to optimize their remarkable properties in the extraction of plant materials offer significant environmental advantages and health and safety benefits over traditional processes for the production of very high quality natural fragrant oils and flavor extracts.

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Reference

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