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THE FIRST TRULY INDEPENDENT WATCHDOG FOR THOSE WORKING WITH NATURAL AROMATIC MATERIALS

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Please find set out below a collection of Cropwatch features (2008-9) on Oakmoss/Treemoss regulatory issues, which raise serious questions about vested interest and the decisions of so-called 'expert' committees.

Please also see Cropwatch's: "Towards a Cropwatch 'Fragrant Lichen' Bibliography: [Oakmoss/Treemoss/Cedarmoss etc.]"

1. Sale / use of fragrant lichen commodities to become virtually illegal in Europe?

Adapted from *Cropwatch Newsletter* (Aug 2008) by the Cropwatch Team (with considerable help from those who have to remain anonymous).

Executive summary.

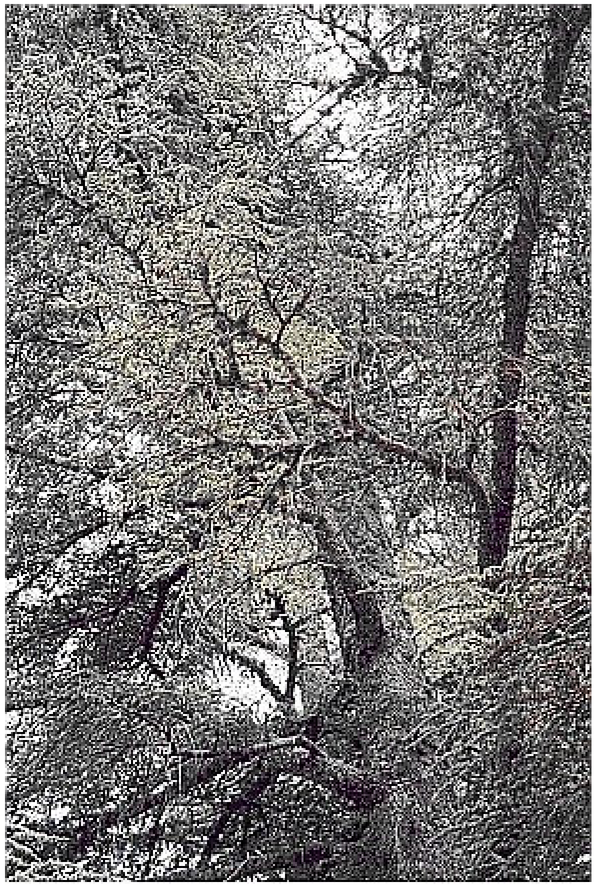
SCCP Opinion SCCP/1131/07 'Opinion on Oakmoss/Treemoss' adopted at the 15th Plenary Meeting on 15th April 2008 limits the potent sensitisers atranol & chloroatranol to 2ppm in oakmoss & treemoss (and cedarmoss) products. These SCCP proposal limits are currently unachievable by industry, reported elsewhere as being the result of a mistaken manufacturers claim. Further, they contrast with a forthcoming IFRA Purity Standard (shortly to be introduced under the 43rd IFRA Amendment) which proposes an achievable limit of 100ppm for atranol & chloroatranol respectively. The SCCP proposal drives a stake right through the heart of perfumery art, heritage & culture, since fragrant moss (lichen) extracts are the cornerstones of both the chypre & fougère accords, so important throughout the history of perfumery. It remains to be seen whether this SCCP Opinion will be transformed into an EC edict, and therefore whether fragrant moss products have any future in Europe. Once again, the SCCP, with its impractical Opinions, is in danger of putting European aroma companies at a disadvantage in the global marketplace, unless the Commissioner can be persuaded otherwise.

Oakmoss: importance in perfumery.

Oakmoss products have a solidly established place in perfumery, being the cornerstone of two renowned accords; the ***chypre*** and the ***fougère***. In the classical *chypre* accord, oakmoss is blended with patchouli, labdanum and other woody animalic and ambery notes, and also often with bergamot. These combinations are the foundation of a family of several leading fine fragrances: Chypre (Coty 1917), Mitsouko (Guerlain 1919), Miss Dior (Christian Dior 1919), Ysatis (Givenchy 1984) & in the male category Aramis (Aramis 1965) & Macassar (Rochas 1980) to name but a few. In the *fougère* accord, striking examples of which include Fougère Royal (Houbigant 1882), Drakkar Noir (Guy Laroche 1982), and Jazz (Yves St. Laurent 1998), oakmoss is blended with coumarin, lavandaceous notes and often with salicylates. Oakmoss products also find uses in colognes, pine fragrances, Crepe de Chine, oriental and fantasy bases etc. etc. (Burfield 2000).

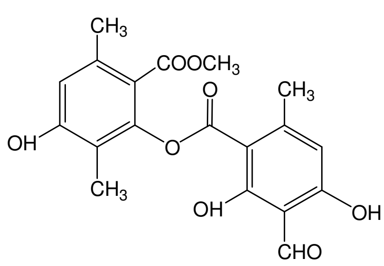
The importance of oakmoss in our perfumery heritage cannot be understated - indeed what are we left with if fragrant moss products are taken away (as nitro-musks and oakmoss have been, in Guerlain's controversially reformulated Mitsouko fragrance, now a shadow of its former self). Well, we are left with a few synthetic oakmoss chemicals, such as *Evernyl* (methyl 2-4-dihydroxy-3-6-dimethylbenzoate) and formerly, the less popularly-utilised *Orcinyl-3* (3-methoxy-5-methylphenol), which the hype from synthetic aroma chemical producers would try to persuade you 'represent the essential character compound of oakmoss'. But, as any practicing perfumer will tell you, there is no way that any single oakmoss synthetic can offer the richness, full body and presence of authentic oakmoss commodities in use, nor approach their superior fixative properties, nor can they duplicate approach the way that oakmoss can radiate and resonate through the entire body of a fragrance. It should also be mentioned that a range of commercial oakmoss products exists, some offering a warm, leathery-mossy character, whilst others offer have woody, mossy - almost marine-like aspects. When materials like oakmoss extracts are restricted by the exiting culture of toxicological imperialism on dubious safety grounds (and this applies also to other vital perfume ingredients such as coumarin and citrus oils - see elsewhere), the 'art of the possible' in perfumery' dies back even further, with a result that fragrance companies, instead of vigorously opposing regulatory change, end-up producing cheap, conformist and essentially poor-quality perfumes with little consumer re-purchase potential, for a increasingly non-discerning market slot.

It is also important to remember, as Joulain (2002) pointed out, that many in the US did not distinguish botanically between the lichen sources of oakmoss (*Evernia prunastri* (L.) Arch), and the source for treemoss & cedarwood (*Pseudevernia furfuracea* (Fr.). This may account for the confusion on various perfume blog sites which have discussed the exact identity of the listed fragrant lichen ingredients employed in a number of classic fragrances, although, to be fair, it should also be remembered that previously oakmoss extracts have invariably been extended with synthetics, and mixed in with treemoss extracts either intentionally, or unintentionally when harvested together..



Cedarmoss growing on *Cedrus atlantica* in High Atlas, Morocco.
Picture: T. Burfield

Oakmoss products have been identified amongst the most frequent fragrance contact sensitizers (Schnuch *et al*. 2007), although the exact chemical identity of the major allergens has been elusive. Gonçalo (1988) for example, considered that that the major sensitisers in oakmoss included atranorin, followed by usnic, evernic and fumarprotocetraric



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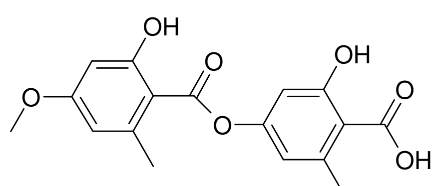

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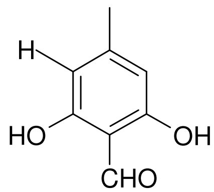
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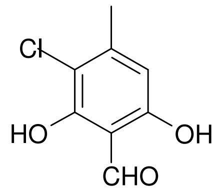
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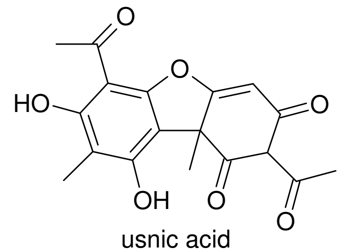

usnic acid

acids, but Bernard *et al*. (2003) note that sensitivity to oakmoss has been associated with components which hold the phenylbenzoate molecular fragment in common, including atranorin & evernic acid. In particular, Bernard *et al.* identified atranol & chloroatranol as strong elicitants in most patients sensitized to oakmoss, and that the oakmoss character compound methyl β-orcinol carboxylate (Evernyl) is also capable of eliciting a reaction in most patients.

How widespread is our exposure to atranorin and other lichen sensitizers? Atraric acid, produced by the hydrolysis of atranorin, has been found in low concentrations of the heartwood from oak species *Quercus robur* & *Quercus petraea* used for staves in the production of **oak barrels used for storage of wine & sprits** (Bourgeois *et al.* 1999). It is likely that colonizing species such as *Parmelia olivetorum* and/or *P. perlata* produce depsides in the wood leading to atranorin accumulation.

It is also worth mentioning, as many of the more travelled amongst you will know, that according to some estimations, up to 1,000 tons/year of *Parmelia nepalensis* (Taylor) Hale ex Sipman is processed into lichen oil, absolute or extract in Western Nepal, and exported for global perfumery and incense use (although the lichens are also used in traditional systems of medicine). Other species such as *Parmelia tinctorum* Delise ex Nyl. & *Usnea* spp. may be co-gathered at the point of harvesting. Moxham (1986) notes the use of *Parmelia nepalensis*, *P. nilgherrensis*, *Ramalina subcomplanata* & *Usnea lucea* in India. Kumar & Muller (1999) have identified the depsides atranorin & diffractaic acid in *Parmelia nepalensis* & *Parmelia tinctorum* extracts. (N.B. note that *Parmelia furfuracea* is a synonym for *Pseudervenia furfuracea*).

A brief regulatory history of fragrant lichen products.

1. An IFRA Standard was introduced for oakmoss extracts in April **1991**; the updated IFRA Standard (2001) limits oakmoss extracts to 0.1.% concentration for finished cosmetic products either left on or washed off the skin, but if oakmoss products are also present in the preparation, the combination of both must not exceed 0.1%. As the presence of resin acids seem to be unavoidable in oakmoss products, IFRA `also imposes an interim limit of 0.1%`dehydroabietic acid for oakmoss extracts.

[*The forthcoming 43rd IFRA Amendment (2008) will introduce a QRA-based system of concentration limits for oakmoss extracts across 33 different fragrance product categories. Purity criteria for oakmoss products are also introduced in the*

43rd IFRA Amendment in the form of limitations on the concentrations of the strong sensitizers, atranol & chloroatranol to 100ppm each].

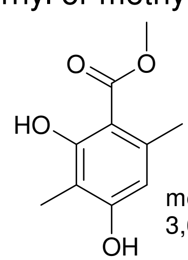
2. The existing IFRA Standard (**1991**, 2001) limits treemoss extracts to 0.1.% concentration for finished cosmetic products either left on or washed off the skin, but if oakmoss products are also present in the preparation, the combination of both must not exceed 0.1%. The IFRA Standard limits dehydroabietic acid (DHA) to 0.8% in treemoss extracts as a marker of 2% of total resin acids, determined by a routine analytical method using HPLC Reverse Phase -Spectrofluorimetry method apparently available from IFRA, according to their website.

[*The forthcoming 43rd IFRA Amendment (2008) will introduce a QRA-based system of concentration limits for treemoss extracts across 33 different fragranced product categories*].

3. Under the 5th EC Framework Program. the EC launched a Quality of Life Initiative & Management of Living Resources key action (**1999**) which included a study of "*Fragrance chemical allergy: a major environmental and consumer health problem in Europe*" Contract No: QLK4-CT-1999-01558 (copy available from Cropwatch in case of difficulty locating it). This project led by J..P Leppoittevin employed a number of leading institutions & scientists in the field, including I.R. White (chairman of SCCP) & S.C. Rastogi (member of SCCP). This project completed in March 2003 at total cost of cost of 1, 927,280 Euros; the major part found by the EU. The project included the development and validation of a method for the identification of sensitisers in complex mixtures using the model of oak moss.

4 The SCC(NF)P at its 14th plenary meeting (24 October **2000**) accepted an Opinion (SCCNFP/0421/00) concerning Oakmoss/Treemoss, that "*... oakmoss/treemoss extracts, present in cosmetic products, have a well-recognised potential to cause allergic reactions in the consumer as fragrance ingredients...*" The Opinion can be seen in full at http://ec.europa.eu/health/ph_risk/committees/sccp/docshtml/sccp_out124_en.htm

5. A scientific paper identifying atranol & chloroatranol as strong elicitants in most patients sensitized to oakmoss was released by Bernard *et al.* (**2003**). Methyl β-orcinol carboxylate (= Evernyl or methyl atrarate),



methy 2,4-dihydroxy-
3,6-dimethylbenzoate

a principle odourant of oakmoss absolute, was also identified as an elicitant in most oakmoss sensitized patients. One of the paper's authors (S.C. Rastogi) is an SCCP committee member.

6. A scientific paper by Bossi *et al.* (**2003)** describes the analysis of atranols in perfumes, employing LC-MS-MS with electrospray ionization (ESI) in negative mode One of the authors (S.C. Rastogi) is an SCCP committee member.

7. A scientific paper Rastogi *et al*. (**2004**) describing the analysis of 31 commercial perfume found that half the perfumes, & some eau de toilettes contained significant amounts of atranol & chloroatranol.. The author, S.C. Rastogi, is an SCCP committee member).

8. Filho *et al* (2004) comment that the present volume of lichen extraction is (ecologically) irreversible given the slow growth of lichen.

9. The SCCP at its 2nd plenary meeting (7 December **2004**) accepted an Opinion (SCCP/0847/04) on atranol and chloroatranol present in natural extracts (e.g. oakmoss and treemoss extracts) with the conclusion: "…*Chloroatranol was shown to cause elicitation of reactions by repeated open exposure at the ppm level (0.0005%) and at the ppb level on patch testing (50% elicit at 0.000015%.). As chloroatranol and atranol are such potent allergens (and chloroatranol particularly so), they should not be present in cosmetic products."* ***Cropwatch comments***: We understand that the robustness of certain parts of the scientific evidence in this Opinion is being queried...

S.C. Rastogi & I.R. White were listed as member & chairman respectively of the above SCCP committee.

10. SCCP Opinion SCCP1131/07 (15th April **2008**) describes an achievable reduction of atranol & chloroatranol in oakmoss extracts to <2ppm according to an analysis method (which is not disclosed), by two different preparative methods (details of which are not disclosed either). S.C. Rastogi and I.R. White were listed as member & chairman respectively of the SCCP committee. .***Cropwatch comments*:** Data from LLNA tests is set out in the Opinion which **did not show** that reduction of atranol & chloroatranol contents in treated (but un-analysed) lichen extracts (of unknown purity) affected the LLNA results. Not withstanding this setback which the SCCP puts down to interlaboratory variation (!), the Opinion calls for a 2ppm limit for atranol & chloratranol in fragrant lichen products which would represent a level **50 times lower** than the proposed IFRA Standard in its forthcoming 43rd Amendment. Evidence showing that treemoss & cedarmoss extracts deserve to be treated in a similar manner to oakmoss extracts is lacking, and Joulain (2002) has previously argued that the principal sensitisation risks in treemoss absolute prepared as described in his presentation, may centre around 7-oxo-dehydroabietic acid (from oxidized pine resin), rather than the atranols. The SCCP's acceptance of claims for achievable atranol & chloroatranol concentrations of <2ppm in treated lichen extracts, via unidentified process(es) from an unidentified source, is typical of the secrecy which pervades the working of Brussels' committees, and we believe contravenes the existing EU guidelines for transparency. Further, contrary

information on this matter has been widely circulated in the trade (e.g. in a perfumery professional members communiqué seen by Cropwatch), which alludes to a mistaken claim made by an unidentified company who directly or indirectly have submitted data to the SCCP. This data, we are lead to believe, initially indicated that the 2ppm limit for atranol & chloratranol was achievable. The company involved however, was reported to have later **withdrawn the claim** when it realised that this experimental research result was not reproducible in a commercial production scenario. Who knows where the truth in this matter lies, but because of the lack of Brussels transparency, Cropwatch can only assume that the SCCP had already seized upon the 2ppm level finding, and, over-hasty as ever, have wrongly adopted the 2ppm limiting level for each sensitizer in the SCCP/1131/07 'Opinion on Oakmoss/Treemoss adopted at the 15th Plenary Meeting on 15th April 2008). If Cropwatch has misunderstood any of the above facts, we can point to the fact that much of the aroma trade has as well. As it is, the situation appears to be a complete shambles.

The Commission is of course not obliged to act on an SCCP Opinion, but as close followers of Brussels affairs will know, finding reverse gear in these situations has proven to be an unlearnt skill, and although they make enough mistakes, we have yet to see the SCCP 'experts' actually own up to an error. As it is, the result may end up in the effective banning of fragrant moss usage in European cosmetics, unless industry lobbyists can persuade the Commissioner otherwise. .

Further background.,,,,,

Interestingly, the scientific literature is pretty scanty on the details of fragrant lichen processing & chemistry, and for this reason Cropwatch has started a fragrant lichen bibliography, in the *Cropwatch Files* section of its website at http://www.cropwatch.org/oakmossbib.htm. This file will be continually updated - contributions, suggestions, & corrections should be directed please to info@cropwatch.org

Oakmoss absolute (Mousse de chêne), concrete, resinoid etc. are derived from the lichen *Evernia prunastri* (L.) Arch. (Fam. Usneaceae) which grows mainly on the bark of oak trees, but also to some extent on spruces & pine trees. Nine thousand tons of oakmoss lichen is gathered annually in S. Europe, in France (formerly in the forests around Fontainebleau), as well from Calabria, Bohemia, Morocco, Algeria, and the area of former Yugoslavia & Bulgaria (Burfield 2000); however this figure may be overstated – Joulain (2002) mentions a figure of 3,000 tons, and Huneck (2001) reported that for the year 1997, 1900 tons of *Pseudevernia furfuracea* and 700 tons of *Evernia prunastri* were processed at Grasse...Some harvested *E. prunastri* lichen has been co-gathered with the lichen *Pseudevernia furfuracea* throughout Europe, but other accounts relate that *Striata pulmonacea, Usnea ceratina, Ramalia farinacea, Ramalia fraxinae, Ramalia pollinaria* and *some Alectoria and Parmelia* species are also mixed in from batch to batch (Burfield 2000). Chinese oakmoss, similar in quality to

European oakmoss, is reportedly produced from *Evernia mesomorpha*, and is also commercially available.

Oakmoss preparation. Oakmoss lichen is not fragrant of itself, and it is only the solvent processing operation which generates the fragrant artifacts which give oakmoss its perfumery value. Ironically then, it cannot be classified as a 'natural product' according to the many bodies now attempting a definition of this term for the cosmetics trade, as recently reported on the *Aromaconnection* website.

Preparation of oakmoss concrete is via solvent hydrocarbon extraction (formerly benzene, but nowadays more often cyclohexane, or hexanes, although acetone and other solvents has been used). of the dried, or freshly wetted dried lichen. Resinoids have been obtained historically by hot solvent extraction, and fragrance synthetics have invariably been added in. Formerly, benzene extracted resinoids had found popular use in soap perfumery. Absolutes can be made directly from the concrete, or by refluxing benzene or hexane extracts with alcohol, and hot filtering out the insoluble material - removal of the alcohol give the absolute in 30-60% yield. Diluting the alcohol extract down to 80% and filtering may give a more soluble product with fewer residues, but further ethanol treatment may give a turbid extract, which when mixed with a saturated salt solution and solvent extracted (benzene was formerly used) further amounts of useful product can be obtained. The identity of the alcohol used will determine the odour - methanol gives sweeter smelling esters, and ethanol produces a sharper smelling product.

Oakmoss incolore and molecular distilled grades of oakmoss are also commercially available. Worryingly, Pybus & Sell (1999) state "with some particularly viscous concretes such as those from oakmoss or treemoss, it is more usual to dissolve the concrete in a high boiling solvent, such as bis-2-ethylhexyl phthalate, and then co-distil the product with the solvent." Hopefully, with current public disquiet over the potential toxicity of phthalates, this practice has been discontinued.

Oakmoss chemistry. As a preface to this section, it may be pertinent to recall Joulain's (2002) remarks to the effect that although the literature reveals qualitative information about oakmoss composition, there is little quantitative data. The fragrant compounds in oakmoss are generated by the degradative action of the solvents on the naturally occurring depsides in the moss (depsides are phenolics composed by two or three monocyclic units linked by an ester bond and derived from orsellinic acid), generating (volatile) odourous monoaryl substances. The character impact compounds of oakmoss are considered to include methyl β-orcinol carboxylate (methyl atrarate, Evernyl)) which imparts a powdery-mossy note, the monomethyl ether of β-orcellinic acid, methyl & ethyl everniate, and the phenolic compounds orcinol and β-orcinol. Boelens (1997) tabled the various yields from different solvent process (extraction and transesterification of the depsides) for both oakmoss and treemoss, reporting that the odour of oakmoss was preferred to treemoss by an odour panel of expert perfumers. Methyl β-orcinol carboxylate was the chief component of both oakmoss absolute and treemoss absolute products (47% and 57% respectively). The quantities of 3-chloro-2,6-dihydroxy-4-methylbenzoate (***Cropwatch comments***: this is possibly a misprint in the original article – surely it should have been 3-chloro-2,6-dihydroxy-4-methylbenzaldehyde, or chloroatranol - 10%), 2,6-dihydroxy-4-methlbenzaldehyde (- atranol - 5% and 6% respectively) and methyl 2,4-dihydroxybenzoate (0.5% and 0.7%) were similar. Oakmoss absolute was found to contain had twice as much cembrene (2%) as treemoss absolute. A full account of the work and composition of the benzene extract and benzene/methylanol transesterification products of oakmoss can be found in an earlier paper by Boelens (1993).

Treemoss (Mousse d'arbre) Treemoss derivatives (concretes, absolutes) are mainly prepared from the lichen species *Pseudevernia furfuracea* (L.) Zopf. with *Usnea barbata, Parmelia sulcata* and other species often co-gathered in. These tree lichens can both be found living on the barks of firs and pines in Southern and Central Europe including and France and Morocco, & Balkan countries, including former Yugoslavia. Preparation of fragrant treemoss products is carried out in a similar manner to the preparation of oakmoss products, although evidence that isopropanol may be included as a processing solvent is shown by the presence of isopropyl haematommate (which does not exist in lichens) in the analysis of the weakly acidic factions of treemoss absolute (Endo *et al.* 1999). It should be noted that Treemoss products are generally considered inferior to oakmoss products and command a lower purchasing price.

Tabacchi (1983) acknowledged that pine products are co-gathered with treemoss, and this has caused the sensitising properties of treemoss extracts to be mis-interpreted by toxicologists at that time. More specifically, Joulain (2002) describes work confirming previous observations by Tabacchi that biosynthesized diterpenoid acids from *Pinus sylvestris* hosts migrate into the oakmoss lichen and assist in their oxidation. The author cites a patented process for producing a treemoss absolute with a low resin acids content but still containing high levels of atranol (0.31%) and chloroatranol (0.15%), which produced no adverse effects at 3% in a suitable solvent in a HRIPT test carried out according to the Marzulli-Maibach protocol with 158 volunteers. Joulain suggested this supported the hypopthesis that 7-oxo-dehydroabietic acid is one of the main sensitizers in treemoss extracts, and that whereas 4-10% of atranorin & chloroatranorin may be present in many treemoss concretes, during the production of absolutes, alcoholysis of the depsides in hot ethanol reduces their content to a level such that they are undetectable by HPLC.

Cedarmoss qualities are derived from *Pseudevernia furfuracea* Ach. growing on the Atlas cedarwood tree *Cedrus atlantica,* found mainly in the Atlas Mountains of Morocco. Solvent extraction produces the resinoid (cyclohexane is used as solvent by some manufacturers), followed by distillation to produce an 'absolute' although other methods for obtaining the absolute are used. Often sweeter than corresponding oakmoss products, it is used in similar perfumery applications.

IFRA Standards for cedarmoss extracts apply exactly as for treemoss extracts, the logic being that they both originate from *Pseudevernia furfuracea* (Fr.). Because it may not be collected exclusively on cedar trees, cedarmoss invariably contains pine twigs and wood fragments which affect the properties & odour of the ingredient.

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2. The Oakmoss & Treemoss Saga – Slight Return.

- from *Cropwatch Newsletter* Jan 2009.

D. Joulain & R. Tabacchi, two people who, perhaps more than any others, have been responsible for unraveling the chemistry of oakmoss & treemoss products over their working lives, have written a review of oakmoss planned to be published in *Flavour and Fragrance Journal* by mid-February 2009. This will be followed by a review on treemoss products by the same authors in the following edition. A third article reviewing the biological properties of lichen products by different authorship is planned for the following edition of the journal. We will be interested to see whether those members of the SCCP with a lesser knowledge & no industrial experience of these lichen products will be embarrassed by the content & conclusions of these articles. You will remember that the SCCP cobbled together an over-hasty SCCP Opinion on limits for the potent sensitizers atranol & chloroatrtanol, which was previously reviewed in the September 2008 edition of *Cropwatch Newsletter*).

3. Excerpt from: 'Safety & Essential Oils– A Catch-Up'.

by Tony Burfield Jan 2009 – to be featured in *Aromatherapy Today* (in press).

……4. **Oakmoss**/treemoss investigations under SCCP/1131/07 are currently the subject of some considerable industrial unrest, concern & heated disagreement. Issues have emerged surrounding missed scientific evidence by SCCP (- not again, you might think – why can't they employ a teenager with computer access to Google, to do a proper literature search?), and there are unanswered questions about partiality of two individual SCCP members, who were also paid researchers on this and other safety topics. Although fragrant lichen products are not central to aromatherapy practice, some very serious questions about the conduct of scientific committees who input into trans-national safety legislation in relation to natural aromatic products are raised by this issue, especially where committees would seem to be acting as witness, judge and jury. The topic can be followed in detail in the *Cropwatch Newsletter* issues Aug 2008 & Jan 2009 and by consulting Cropwatch's fragrant lichen products bibliography on the Cropwatch website, but stand-by, there is more to come!