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Document Cover Sheet

<https://www.ams.usda.gov/rules-regulations/organic/petitioned-substances>

Document Type:

**National List Petition or Petition Update**

A petition is a request to amend the USDA National Organic Program's National List of Allowed and Prohibited Substances (National List).

Any person may submit a petition to have a substance evaluated by the National Organic Standards Board (7 CFR 205.607(a)).

Guidelines for submitting a petition are available in the NOP Handbook as NOP 3011, National List Petition Guidelines.

Petitions are posted for the public on the NOP website for Petitioned Substances.

**Technical Report**

A technical report is developed in response to a petition to amend the National List. Reports are also developed to assist in the review of substances that are already on the National List.

Technical reports are completed by third-party contractors and are available to the public on the NOP website for Petitioned Substances.

Contractor names and dates completed are available in the report.

# Tocopherols

## Handling/Processing

### Summary of Petitioned Use

This limited scope technical report provides updated information to the National Organic Standards Board (NOSB) to support the sunset review of tocopherols, listed at 7 CFR 205.605(b)(36). This limited scope technical report focuses on the manufacturing processes used to make tocopherols, which are allowed as synthetic nonagricultural substances for use in organic handling. We also discuss organic agricultural products that may be used in place of tocopherols.

Tocopherols are allowed for use in organic processing and handling, as ingredients or processing aids. According to the annotation at § 205.605(b)(36), tocopherols must be derived from vegetable oil when rosemary extracts are not a suitable alternative.

Tocopherols were originally reviewed by the NOSB in 1995 (NOSB, 1995). They were included on the National List of Allowed and Prohibited Substances (hereafter referred to as the “National List”) with the first publication of the National Organic Program (NOP) Final Rule (65 FR 80548, December 21, 2000). The NOSB has continued to recommend the renewal of tocopherols in 2005, 2011, 2015, and 2019 (NOSB, 2005, 2011, 2015, 2019).

In 2016, the NOSB Handling Subcommittee considered adding tocopherols to § 205.605(a), as an allowed nonagricultural nonsynthetic substance in organic handling, while simultaneously maintaining the listing at § 205.605(b) (NOSB, 2016). The subcommittee supported this action through a unanimous vote (NOSB, 2016). However, the full board returned the proposal to the subcommittee for further work (NOSB, 2016). In 2017, the Handling Subcommittee decided to postpone adding tocopherols to § 205.605(a) until gathering public comment (NOSB, 2017). One member of the subcommittee noted that it wasn’t necessary to list tocopherols at both § 205.605(a) and § 205.605(b) (NOSB, 2017). They also voted to update the annotation for tocopherols to read: “Derived from plant oils. Non-synthetic or organic tocopherols are to be used when commercially available” (NOSB, 2017). The Handling Subcommittee then decided to defer further work on tocopherols (NOSB, 2017).

### Evaluation Questions for Substances to be used in Organic Handling

**Evaluation Question #1: Describe the most prevalent processes used to manufacture or formulate the petitioned substance. Further, describe any chemical change that may occur during manufacture or formulation of the petitioned substance when this substance is extracted from naturally occurring plant, animal, or mineral sources [7 U.S.C. 6502(21)].**

Tocopherols exist naturally in plant tissues as D-alpha-, D-beta-, D-gamma, and D-delta-tocopherol (Moreau & Lampi, 2012; Yui et al., 2016). These forms have mirror centers (chiral centers) at the 2<sup>nd</sup>, 4<sup>th</sup>, and 8<sup>th</sup> carbon position (Yui et al., 2016). This spatial arrangement is abbreviated as (R,R,R) and means that the atoms at these centers are arranged in a clockwise rotation. The seven alternate forms (which do not occur naturally in plant tissue), including a commercially common DL-alpha-tocopherol, provide alternate biological activities due to the differences in spatial arrangement (i.e. counterclockwise atomic arrangement) (Moreau & Lampi, 2012; Yui et al., 2016).

The main sources of tocopherols are plant derivatives (such as various ground meals) and, more commonly, the deodorized distilled sludge (DD) obtained from conventional vegetable oil refining (Liu et al., 2019; Vafaei et al., 2022).

50 Common plant sources of tocopherols are (Malekbala et al., 2017; Vafaei et al., 2022):

- 51 • grape seeds
- 52 • almond seeds
- 53 • olive tree leaves
- 54 • palm leaves
- 55 • olive husks
- 56 • wheat germ
- 57 • walnuts
- 58 • palm oil
- 59 • rice bran oil
- 60 • soybean oil
- 61 • soy oil
- 62 • peanut oil
- 63 • sunflower oil
- 64 • DD

65

66 To extract tocopherols, manufacturers use different methods, depending on the source material and  
 67 available resources and instruments (Saini & Keum, 2016). The primary extraction methods used by  
 68 manufacturers are:

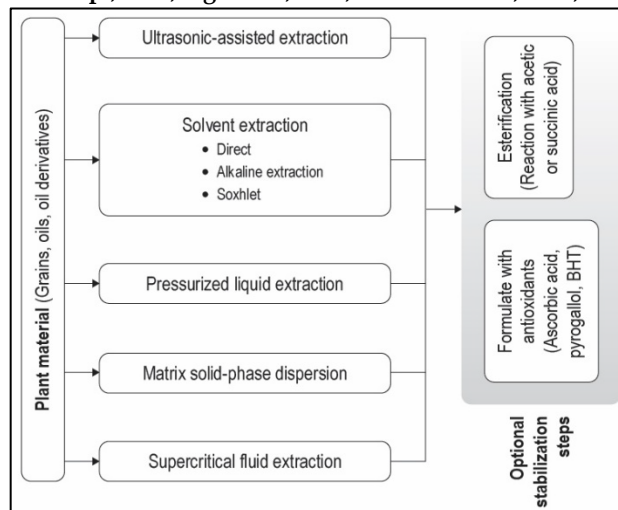
- 69 • solvent extraction
- 70 • pressurized liquid extraction
- 71 • extraction using matrix solid phase dispersion
- 72 • supercritical fluid extraction
- 73 • ultrasonic assisted extraction
- 74 • molecular distillation

75

76 These methods are described in more detail, below (also, see [Figure 1](#), below). Production methods for  
 77 tocopherols are very complex and can involve variations in solvents, acids and bases, and additives such as  
 78 stabilizers. It is therefore not feasible for us to identify and explain all of the possible production variations.  
 79 Consequently, we also cannot easily classify all sources of tocopherols later in [Evaluation Question #2](#).  
 80 Material reviewers will likely need to review detailed manufacturing processes of individual tocopherol  
 81 products on a case-by-case basis, evaluating them against Guidance NOP 5033-1 *Decision Tree for*  
 82 *Classification of Materials as Synthetic or Nonsynthetic* (NOP, 2016a) and NOP 5033-2 *Decision Tree for*  
 83 *Classification of Agricultural and Nonagricultural Materials for Organic Livestock Production or Handling* (NOP,  
 84 2016b).

85

86 **Figure 1. Production of tocopherols from sources other than DD. Sources: Jafarian Asl et al., 2020; Malekbala et al.,**  
 87 **2017; Moreau & Lampi, 2012; Ogonna, 2009; Saini & Keum, 2016; Vafaei et al., 2022.**



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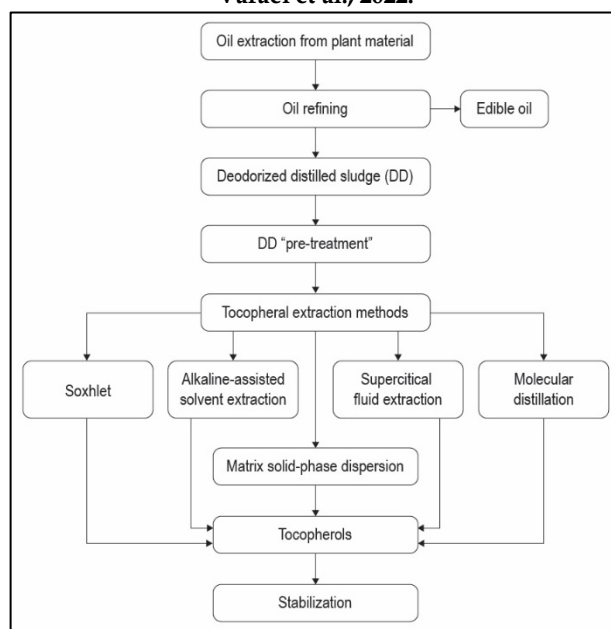
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90 *Deodorized distilled sludge as a source for tocopherols production*

91 Before we discuss the aforementioned extraction methods related to other plant sources (e.g. extraction  
92 from grape seeds or olive husks), we describe here the manufacturing process of tocopherols from DD,  
93 which is likely the most common source of tocopherols (Liu et al., 2019; Vafaei et al., 2022). Extraction of  
94 tocopherols from DD also involves the use of extraction techniques described in other sections.  
95

96 When edible oils are refined, desirable tocopherols are unintentionally separated out with waste materials.  
97 The “DD” process we describe here recovers tocopherols from the waste sludge (see [Figure 2](#)).  
98

99 **Figure 2. Production of tocopherols from deodorized distilled sludge. Sources: da Silva et al., 2023; Gharby, 2022;**  
100 **Vafaei et al., 2022.**



101  
102

103 Manufacturers produce edible oils in two steps (Gharby, 2022; Vafaei et al., 2022):

- 104 1. They first extract oils from the seeds (mechanically or by organic solvent).
- 105 2. Then they refine the crude oil.

106

107 They convert the crude oil to refined oil using physical and chemical refining stages (Gharby, 2022).

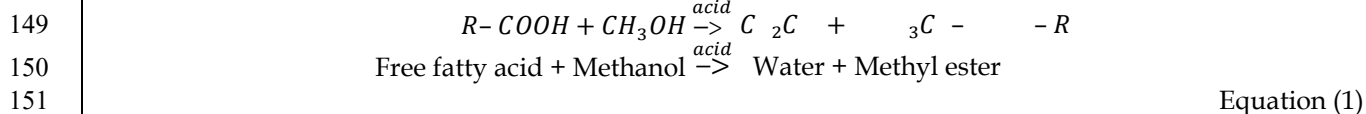
- 108 3. The oil is degummed to remove phosphatides. This can happen chemically, using phosphoric or  
109 citric acid, or by using the enzyme, phospholipase C.
- 110 4. The oil is then neutralized to remove free fatty acids, phospholipids, metals, and chlorophylls. This  
111 can happen chemically using sodium hydroxide to form a soap stock (insoluble in oil and  
112 physically removed) or via steam distillation (see the last, deodorize step).
- 113 5. Next, the oil is washed and dried, if applicable. This removes residual soaps and water present  
114 from the neutralization step.
- 115 6. The oil is bleached to remove colored pigments and residues (phosphatide, soap, phospholipid  
116 contaminants, lipid peroxidation products, other impurities). Adsorption bleaching clays, activated  
117 carbon, specialized silica, or a combination of these remove the pigments and residues.
- 118 7. The oil is dewaxed (winterized). This step is applicable to wax-rich oils like corn germ oil and  
119 sunflower oil. Chilling (10–15 °C) of oil for several hours crystallizes the waxes. The cooled oil is  
120 filtered to separate crystallized wax from the vegetable oil.
- 121 8. Lastly, the oil is deodorized to remove free fatty acids and other volatile compounds. Vacuum  
122 steam distillation (180–240 °C, 2–8 mmHg) removes the undesirable compounds, along with other  
123 more valuable substances such as some (but not all) tocopherols. This sludge becomes the DD  
124 material, which is further processes to isolate free alpha-tocopherols.  
125

126 DD contains around 30–40% of the tocopherols (tocopherols and tocotrienols) available in the oil (Liu et al., 2019;  
 127 Vafaei et al., 2022). Soybean oil deodorized distillate is the primary source of tocopherols due to soybean  
 128 oil's low cost (da Silva et al., 2023). However, deodorizer distillates of corn and rapeseed oils are also  
 129 sometimes used (Moreau & Lampi, 2012). DD sources are commercially extracted by various methods (see  
 130 [Table 2](#)).

131  
 132 DDs contain a variety of substances such as tocopherols, tocotrienols, phytosterols, squalene, free fatty  
 133 acids, and acylglycerols (da Silva et al., 2023; Gharby, 2022). In order to separate these substances more  
 134 efficiently from each other, manufacturers perform a “pretreatment” step that esterifies the fatty acids in  
 135 the mixture.<sup>1</sup> Esterification changes the physical properties of the fatty acids, which otherwise would have  
 136 similar molecular weights and vapor pressures to the other compounds present (including tocopherols).  
 137 The goal of this esterification pretreatment is to convert the free fatty acids into alkyl esters [see  
 138 [Equation \(1\)](#), below], thus changing their physicochemical properties (solubility, vapor pressure, and  
 139 boiling point) (da Silva et al., 2023; Liu et al., 2019). This improves the subsequent separation of the  
 140 compounds (da Silva et al., 2023). The pretreatment process is as follows (da Silva et al., 2023):

- 141 1. DD is heated between 40–80 °C in a reactor.
- 142 2. Separately, an acid catalyst (e.g., sulfuric acid) is slowly dissolved in a short-chain alcohol (e.g.,  
 143 methanol or ethanol).
- 144 3. The acid-alcohol mixture is added to the liquefied DD.
- 145 4. The mixture then refluxes with condensers at 15 °C.<sup>2</sup>
- 146 5. Unreacted alcohol is vacuum distilled (80 °C for 40 minutes in the case of methanol).
- 147 6. The catalyst is removed by washing with distilled water until the wash water reaches pH 7.

148



152

153 The end-product is a methyl (or ethyl) esterified deodorizer distillate (MEDD) (da Silva et al., 2023), which  
 154 can then be used in one of the following tocopherol extraction methods (discussed later):

- 155 • Soxhlet extraction
- 156 • alkaline-assisted solvent extraction
- 157 • matrix solid-phase dispersion (MSPD)
- 158 • supercritical fluid extraction
- 159 • molecular distillation

160

161 Some amount of tocopherol fatty acids (tocopherols bound to fatty acids) react with the methanol to create  
 162 tocopheryl quinone and tocopherol esters (da Silva et al., 2023; Kreps et al., 2016). During the vacuum  
 163 distillation step of the pretreatment, the rising temperature creates tocopherol esters and tocopheryl  
 164 quinones as impurities, along with the desired free alpha-tocopherols (Kreps et al., 2017). These impurities  
 165 further degrade to other oxidation products, which cannot be effectively separated from the desired free  
 166 alpha-tocopherol extract. Many of these tocopherol degradation products have very similar properties,  
 167 both to each other and to the desired free alpha-tocopherols. These impurities are created by chemical  
 168 synthesis, and because they remain in the tocopherol mixture, may affect the classification of tocopherols  
 169 from this source (see [Evaluation Question #2](#), below).

170

<sup>1</sup> Esterification is a process where an acid is chemically bonded to an alcohol. In this case, the tocopherol contains a carboxylic acid (COOH) group, which is then bonded to an alcohol such as methanol. Heat and a catalyst are typically needed to drive the reaction.

<sup>2</sup> Reflux refers to a process where liquid is boiled, and the resulting vapor liquefies and returns to the boiling vessel. Condensers are used to help cool the vapors, typically made from wound tubes.

171 *Solvent extraction for tocopherols production*

172 Solvent extraction is the traditional (and most common) tocopherol extraction method from non-DD  
 173 sources (Ogbonna, 2009; Saini & Keum, 2016). The raw sources of tocopherols for extraction are grains, oils,  
 174 and oil derivatives due to their lipid-soluble (hydrophobic) nature.

- 175 1. Source materials are first ground (if solid), and then homogenized by vortexing, sonication or  
 176 ultrasound to improve the extractability.
- 177 2. When fresh fruits and vegetable sources are used, manufacturers freeze dry the source material  
 178 prior to the initial homogenization to avoid degradation that would otherwise occur. Alternatively,  
 179 source materials may be treated with a stabilizing agent such as ascorbic acid, butylated  
 180 hydroxytoluene (BHT), or pyrogallol during the extraction (Saini & Keum, 2016).
- 181 3. The next step is solvent or solvents selection; the most typical solvents include ethanol and hexane  
 182 and may be a combination of solvents (see [Table 1](#), below).
- 183 4. Extraction as described below.

184  
 185 **Table 1: Common tocopherols source-solvent combinations. Sources: Moreau & Lampi, 2012; Saini & Keum, 2016.**

| Source                                       | Solvent                         |
|--|---------------------------------|
| Microwave-oven dried leaves                  | Acetone                         |
| Cereal grains (wheat, rye, barley, and corn) | Methanol                        |
| Oats   | Acetonitrile                    |
| Tree nuts                                    | Chloroform and methanol; hexane |
| Legumes                                      | Hexane                          |
| Palm oil                                     | Methanol and isopropanol        |
| Vegetable oils                               | Isopropanol and chloroform      |
| Green leafy vegetables                       | Methanol and dichloromethane    |

186  
 187 The solvent extraction method depends on the source material, solvent, and analytical method chosen  
 188 (Moreau & Lampi, 2012; Saini & Keum, 2016).<sup>3</sup> Three solvent extraction methods exist:

- 189 • alkaline-assisted solvent extraction (saponification)
- 190 • direct solvent extraction
- 191 • Soxhlet extraction

192  
 193 Alkaline-assisted solvent extraction

194 Alkaline hydrolysis is used in conjunction with solvent extraction (see [Figure 1](#), below) in order to improve  
 195 the extractability of tocopherols (Moreau & Lampi, 2012; Saini & Keum, 2016). Also known as hot  
 196 saponification when performed at high temperature, this method specifically is used as a pre-treatment to  
 197 remove carbohydrate and protein impurities, as well as neutral or other saponifiable lipids that may  
 198 interfere with the chromatographic analysis (Moreau & Lampi, 2012). Hot saponification is typically used  
 199 on hard tissue and high-lipid source materials such as grains and oils, where carbohydrate-lipid or protein-  
 200 lipid interactions need to be broken down prior to extraction (Moreau & Lampi, 2012; Saini & Keum, 2016).

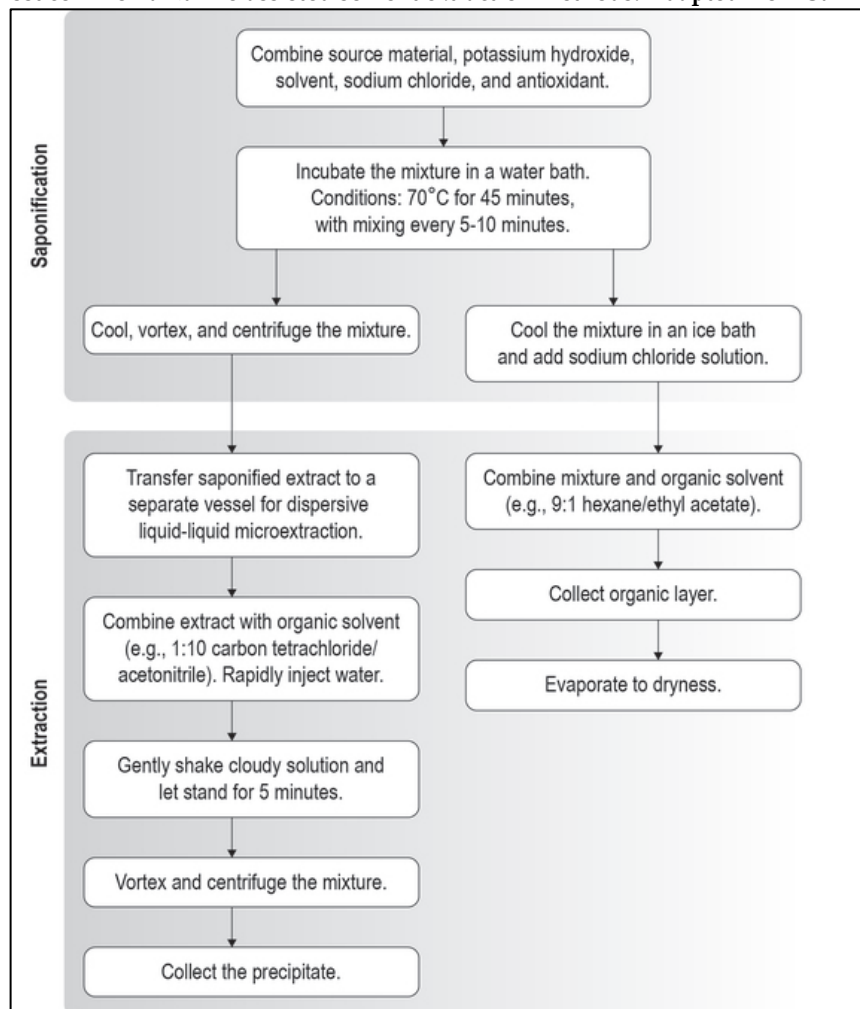
201  
 202 Room temperature saponification is used in leafy source materials. Room temperature saponification is  
 203 used to remove chlorophyll and interfering lipids when the analysis method is by mass spectrometry (Saini  
 204 & Keum, 2016). The hydrolysis process allows for the disintegration of these compounds, leaving an  
 205 isolated lipid-soluble extract.

206  
 207 Solvent extraction directly follows the saponification process. We discuss the effect of saponification in the  
 208 synthetic/nonsynthetic classification of tocopherols in [Evaluation Question #2](#).

209

<sup>3</sup> Analytical method refers to the quality control method used to analyze the final extract.

210

**Figure 3: Most common alkaline-assisted solvent extraction methods. Adapted from Saini et al. (2016).**

211

212

### 213 Direct solvent extraction

214 Direct solvent extraction works by mixing (by vortexing, sonicating, or using ultrasound) the tocopherol  
 215 source, for example leafy green vegetables or legumes, with the chosen solvent (Moreau & Lampi, 2012).  
 216 The solvent is subsequently evaporated. The evaporation temperature and length of time are dependent on  
 217 the solvent.

218

### 219 Soxhlet extraction

220 Soxhlet extraction, or a modified version of direct solvent extraction, is another commonly used method.  
 221 Manufacturers may elect this path to control or reduce the destruction of heat-sensitive compounds within  
 222 the plant material (Jafarian Asl et al., 2020). This path involves a solvent and a silica column, as a  
 223 processing aid (Pavia et al., 2011):

- 224 1. The source material (i.e., spinach) is placed in the extraction chamber on a filter or porous thimble.
- 225 2. Then, a round-bottom flask containing the solvent (i.e., mixture of methanol and dichloromethane)  
 226 is attached below the extraction chamber. A reflux condenser is attached above the chamber.
- 227 3. The solvent is heated to its boiling point. Evaporated liquid condenses, filling up the chamber.
- 228 4. The column drains when full, carrying the extract through the filter and into the flask below. This  
 229 reflux can happen for as many cycles as needed to improve extract concentration and purity.

230

231 The following is a modified version of the Soxhlet extraction (Jafarian Asl et al., 2020). In this method,  
 232 tocopherol-containing oil compounds are introduced into the extractor more gradually and at a lower flow

233 rate, thereby reducing the destruction of heat-sensitive compounds in the plant material (Jafarian Asl et al.,  
234 2020).

- 235 1. The solvent and source material are combined and then mixed as in a direct solvent extraction.
- 236 2. The silica gel is added to the mixture. The polar portion of the source material is absorbed into the  
237 silica gel.
- 238 3. The silica gel and solvent-source mixture are placed in the chamber.
- 239 4. A separate solvent is added for the refluxing step. This new solvent extracts the tocopherols from  
240 the silica gel/first solvent.

241  
242 All of the solvent extraction methods described in this section are time-consuming processes that involve  
243 toxic solvents for the extraction (Saini & Keum, 2016). Manufacturers elect these solvent methods because  
244 of their simplicity and low cost—the main advantage being that the extraction occurs at room temperature  
245 and pressure. There is no need for specialized equipment.

246  
247 There is some limited evidence that maceration may be used along with solvent extraction to isolate  
248 tocopherols (Ozsoz et al., n.d.). The method uses the most solvent and has low extract yields when  
249 compared to the other pathways. Plant material is soaked in an organic solvent, such as ethanol, for 2–10  
250 days at room temperature. Phytochemical compounds, including tocopherols, are directly released into the  
251 solvent. It is not clear if this method is commercially used. The literature does not mention how  
252 manufacturers isolate specific phytochemicals from the solvent macerate (Ozsoz et al., n.d.).

#### 253 254 *Pressurized liquid extraction (PLE) for tocopherols production*

255 Pressurized Liquid Extraction (PLE) is similar to direct solvent extraction, but it increases the pressure of  
256 the system to shorten the extraction time (Moreau & Lampi, 2012). Because of the shorter extraction time,  
257 the method is sometimes referred to as accelerated solvent extraction (ASE). Also, like direct solvent  
258 extraction, a saponification step may occur when the source material is a grain (Delgado-Zamarreño et al.,  
259 2009). Saponification is used to disrupt bonds that form between carbohydrates and lipids, as well as  
260 proteins and lipids.

261  
262 Commonly, manufacturers mix the source material with a drying agent (e.g. diatomaceous earth) prior to  
263 the extraction (Delgado-Zamarreño et al., 2009; Moreau & Lampi, 2012). This aids in the extraction by  
264 removing water from the system. Manufacturers optimize parameters to the solvent, usually methanol or a  
265 mixture of methanol and isopropanol (Moreau & Lampi, 2012).

- 266 • For methanol, the temperature is set to 50 °C at 108.6 atmospheres of pressure (atm) for one 5-  
267 minute cycle.
- 268 • For hexane, the temperature is set to 80 °C at 102 atmospheres of pressure (atm) for one 10-minute  
269 cycle.

270  
271 Compared to direct solvent extraction, this method uses higher temperatures because the pressurized  
272 liquid solvents remain as liquids above their standard pressure boiling points. As pressure increases, so  
273 does the boiling point of the liquids.

274  
275 Manufacturers may elect this method for the enhanced solubility qualities; tocopherols are more  
276 solubilized under this higher pressure and temperature (Saini & Keum, 2016). However, the method is not  
277 suitable for sources where tocopherols exist at low concentrations. Yields are inversely proportional to oil  
278 yields (Moreau & Lampi, 2012).

#### 279 280 *Matrix solid-phase dispersion (MSPD) for tocopherols production*

281 Matrix solid-phase dispersion (MSPD) is a method that uses a dispersion sorbent, such as the porous  
282 surface of alumina, to adsorb tocopherols.<sup>4</sup> It is a labor-intensive and time-consuming method, but it uses  
283 less solvent than solvent extraction (Saini & Keum, 2016). Source material and a suitable dispersion sorbent  
284 are manually blended, then transferred to a column to be eluted with a solvent (Pavia et al., 2011; Saini &

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<sup>4</sup> Sorbent: A general term for materials that can both adsorb (attract to the surface of the material by physical attraction) or absorb (penetrate into the material like a sponge).



285 Keum, 2016).<sup>5</sup> Source material can be plant material like barley, eluted with methanol (Saini & Keum,  
286 2016), or palm fatty acid distillate, eluted with hexane then isopropanol (Malekbala et al., 2017).  
287 Manufacturers using palm fatty acid distillate as a source material will first neutralize the material to  
288 establish a mildly alkali environment, thereby concentrating the tocopherols content (Malekbala et al.,  
289 2017). Heat is applied in this process (60 °C). Neutralizing the material concentrates the content by 5.6  
290 times when compared to the original distillate; subsequent alkali hydrolyzation concentrates the content by  
291 an additional 11.8 times (Malekbala et al., 2017).

292

### 293 *Supercritical fluid extraction (SFE or SCF) for tocopherols production*

294 Supercritical fluid extraction (SFE or SCF) is a widely used method for the extraction of lipids, flavors, and  
295 bioactive compounds (Saini & Keum, 2016). The process works by forcing the solvent into a physical state  
296 between that of a liquid and a gas (Malekbala et al., 2017). The result is a solvent with a higher diffusivity  
297 compared to conventional solvents (e.g., ethanol, hexane, etc.) that is easily removed without leaving  
298 residues behind. A supercritical solvent will have the density of a liquid and dissolve material like a liquid  
299 but behave like a gas and diffuse through solids (Vafaei et al., 2022). The extraction time and rate of source  
300 material deterioration are also lower (Malekbala et al., 2017). The extraction is affected by solvent flow rate,  
301 operating pressure, temperature, amount of modifier, time, and particle size (Xu et al., 2017).

302

303 Carbon dioxide is the preferred and standard solvent because of its inert nature, low supercritical  
304 temperature and pressure (31.1 °C at 73 atm), and non-toxicity (Jafarian Asl et al., 2020; Malekbala et al.,  
305 2017; Saini & Keum, 2016; Vafaei et al., 2022). Manufacturers may elect to combine solvents for efficiency  
306 and cost (Jafarian Asl et al., 2020). A common combination is ethanol at 5 wt% to 95 wt% supercritical  
307 carbon dioxide (SC-CO<sub>2</sub>).

308

309 SFE conditions are near-environmental temperature, ideally 55 °C, but at elevated pressure of 473.72 atm  
310 (Saini & Keum, 2016). The process is described by two major steps (Xu et al., 2017):

- 311 1. The soluble compounds are extracted from the plant material by the supercritical solvent.
- 312 2. The target compound is separated from the supercritical solvent by a rapid pressure reduction  
313 and/or a temperature increase, or both.

314

315 Extract collection happens in a small volume of solvent which later sublimates to a vapor at room  
316 temperature and pressure (Xu et al., 2017). Alternatively, the extract may be directly collected in a solid-  
317 phase trap within the SFE apparatus. There is no cleanup or filtration required. Carbon dioxide may be  
318 recovered from the system without damaging the plant material by reducing the pressure of the system  
319 (Malekbala et al., 2017; Saini & Keum, 2016).

320

321 Manufacturers are limited by equipment expense and its low-volume capacity (Saini & Keum, 2016).  
322 Jafarian Asl et al. (2020) note that the method is not suitable for industrial scale operations because of the  
323 capacity limitations and blockages caused by water in the source material. There is also an added limitation  
324 stemming from SC-CO<sub>2</sub> – carbon dioxide has a limited polar molecule dissolution, even at elevated  
325 densities (Jafarian Asl et al., 2020). It will not dissolve polar substances such as tocopherols as readily as  
326 other extraction solvents (i.e., ethanol). This is the reason why manufacturers see an increase in efficiency  
327 when other supercritical solvents are added to carbon dioxide. Despite these limitations, SC-CO<sub>2</sub> extraction  
328 is commercially used for the recovery of tocopherols from soybean oil deodorizer distillate (Jafarian Asl et  
329 al., 2020).

330

### 331 *Ultrasonic-assisted extraction (UAE) for tocopherols production*

332 Ultrasonic-assisted extraction (UAE) is a family of methods that work by exploiting the cavitation  
333 phenomenon (Xu et al., 2017). Manufacturers move fluids (in this case a solvent or solvents) at high speed  
334 on the surface of a system (the source material) via compression and rarefaction (Xu et al., 2017; Zhang,  
335 2016).<sup>6</sup> The system, as a result of the high speed, is low in pressure (Xu et al., 2017). Continuous

<sup>5</sup> Elute: Collection of adsorbed substance by washing with a solvent.

<sup>6</sup> Rarefaction waves: also known as relief waves, these are waves moving in the opposite direction of the compression (shock wave). These waves spread out in space and continue to spread as time goes on.

336 compression and rarefaction cause disturbances in the system, creating bubbles known as cavitation  
337 bubbles. Over the course of a few cycles, the size of the bubbles grows until they reach a critical point  
338 where the low-pressure bubble implodes because of the high-pressure surroundings. The cavitation bubble  
339 collapse releases large amounts of energy, generating extreme heat (4726 °C) and pressure (1000 atm) at  
340 room temperature. Plant tissue cell walls are destroyed as a result of this energy, thereby facilitating the  
341 release of oils rich in tocopherols. The extraction itself is dependent on the types of solvent used, solvent  
342 volume, source material characteristics (moisture and particle size), ultrasound frequency and intensity,  
343 temperature, and extraction time.

344  
345 Manufacturers may elect a variation of this method in order to avoid tocopherol heat degradation (Saini &  
346 Keum, 2016; Xu et al., 2017). The optimum conditions are 40 °C for 50 minutes (Saini & Keum, 2016). Pulsed  
347 ultrasonic-assisted technique (PUAE) is a variation that turns the ultrasonic processor on and off  
348 intermittently (Xu et al., 2017). This generates less heat than continuous pulsing.

- 349 1. The source material and solvent (e.g., methanol) are placed in a vessel.
- 350 2. The vessel is immersed in an ultrasonication bath for 5–60 minutes. Each cycle is 0.2–0.7 seconds in  
351 length.
- 352 3. The resulting oil with tocopherols is collected and filtered.

353

#### 354 *Molecular distillation for tocopherols production*

355 Molecular distillation is a main method of extraction for DD sources (Liu et al., 2019). The method is  
356 characterized by a short exposure of the distilled liquid to elevated temperatures, a high vacuum (thereby  
357 reducing the amount of oxygen present in the system), and a small distance between the condenser and the  
358 evaporator to minimize the drop in pressure and shorten the amount of time the tocopherols are exposed  
359 to heat. Through this process, DDs are fractionally distilled and collected without the need for solvent.

360

361 To increase yields and purity of the distillation, manufacturers may treat DD sources with caustic methanol  
362 by the following procedure:

- 363 1. The DD source is mixed with hexane.
- 364 2. The mixture is combined with methanol and sodium hydroxide. The mixture separates into two  
365 layers – a tocopherol-containing caustic methanol and an impurity-containing phase.
- 366 3. The caustic methanol phase is mixed with hexane three times.
  - 367 a. In order to prevent the loss of tocopherols, the hexane layers are treated with glacial acetic  
368 acid and sodium hydroxide three times.
  - 369 b. Water is added to separate the mixture into two phases – a tocopherol-enriched phase  
370 (hexane) and a neutralized methanol phase.
  - 371 c. The methanol phase is washed with hexane. The tocopherols remain in the hexane.
  - 372 d. The two layers are combined.
- 373 4. The manufacturer removes the hexane under vacuum distillation. The result is an 82% tocopherol  
374 and 7% sterol mixture.
- 375 5. Tocopherols are then extracted through molecular distillation.

376

377 **Table 2. Summary of tocopherols extraction methods. Sources: Jafarian Asl et al., 2020; Jiang et al., 2006; Liu et al.,**  
 378 **2019; Malekbala et al., 2017; Moreau & Lampi, 2012; Saini & Keum, 2016; Xu et al., 2017.**

| Extraction method             | Source materials                      | Common solvents, reagents, and stabilizers   | Conditions                            |
|-------------------------------|---------------------------------------|--|---------------------------------------|
| Direct solvent                | Plant material, oils, grains          | Methanol, ethanol, hexane, acetonitrile, isopropanol, dichloromethane, acetone, chloroform, ascorbic acid, BHT, pyrogallol             | 20–25 °C, 1 atm                       |
| Soxhlet                       | Oils, DDs                             | Methanol, ethanol, hexane, acetonitrile, isopropanol, dichloromethane, acetone, chloroform, ascorbic acid, BHT, pyrogallol, silica gel | 40–80 °C, 1 atm in solvent chamber    |
| Alkaline hydrolysis assisted  | Oils, DDs, grains                     | Potassium hydroxide, sodium chloride, ascorbic acid, BHT, pyrogallol, hexane, ethyl acetate, carbon tetrachloride, acetonitrile        | 70 °C, 1 atm                          |
| Pressurized liquid            | Spinach, corn, cranberry, pomegranate | Methanol, isopropanol, hexane, drying agents (i.e., diatomaceous earth)  | 50–80 °C, 100–110 atm                 |
| Matrix solid-phase dispersion | Plant material                        | Alumina, methanol  | 20–25 °C, 1 atm                       |
| Matrix solid-phase dispersion | Palm fatty acid distillate            | Alkali material, alumina, hexane, isopropanol  | 60 °C, 1 atm; 20–25 °C, 1 atm         |
| Supercritical fluid           | Soybean oil DD                        | Carbon dioxide, supercritical; ethanol, supercritical  | 55 °C, 473.72 atm                     |
| Ultrasonic assisted           | Plant material                        | Hexane, methanol   | 40 °C, 1 atm                          |
| Molecular distillation        | DDs                                   | None   | 170–230 °C, $2.63 \times 10^{-5}$ atm |

379

380 *Separation methods*

381 Manufacturers may elect to further purify a raw oil containing tocopherols obtained from the above  
 382 methods. SFE, molecular distillation, and adsorption chromatography are popular methods for further  
 383 purification (Malekbala et al., 2017).<sup>7</sup>

384

385 *Stabilization*

386 Manufacturers sometimes take steps to prevent tocopherol oxidation as some amount of the extracted  
 387 tocopherols degrade (Kreps et al., 2017). To this end, manufacturers control the environmental conditions,  
 388 such as by working under subdued light and low temperature, use an inert gas (i.e. nitrogen) in the  
 389 extraction chamber, and apply antioxidants (Moreau & Lampi, 2012). Temperature is the main cause of  
 390 degradation and results in the creation of tocopherol esters (Kreps et al., 2017). Antioxidants such as  
 391 ascorbic acid, pyrogallol, and BHT may be added during the extraction process and not removed from the  
 392 final product (Moreau & Lampi, 2012). The literature notes that this addition is very common, specifically  
 393 for solvent methods.

394

395 Many lipids with free hydroxyl (-OH) groups can form more stable, higher activity esters with molecules  
 396 that contain a carboxylic acid (-COOH) group (Moreau & Lampi, 2012). Manufacturers may intentionally  
 397 elect to convert free alpha-tocopherol, a form of unbound tocopherol, to its more stable ester form by  
 398 reacting the compound with acetic or succinic acid (Moreau & Lampi, 2012; Rich et al., 2012). The end  
 399 product is D- or DL-alpha-tocopheryl acetate or succinate (Rich et al., 2012). We did not find information  
 400 regarding post-extraction steps when non-solvent paths are used. Because degradation is a general concern  
 401 with tocopherols, it may be that additives or acetic/succinic acid treatment are used regardless of  
 402 extraction method.

403

404 More traditional methods used for extracting edible oils use organic solvents, that inevitably leave residues  
 405 (Ogbonna, 2009). Solvents leave residues at the surface of the plant material and need elevated  
 406 temperatures for their removal (Malekbala et al., 2017). This heating can potentially lead to chemical

<sup>7</sup> A silica or alumina column is used to adsorb compounds. Compounds are then separated from the surface of the silica or alumina using a solvent of similar polarity. The solvent is removed by evaporation.

407 transformation of oleoresins present in the material.<sup>8</sup> However, the heating is key to reducing solvent  
408 residues to very small concentrations, at around 25–30 ppm or less (Malekbala et al., 2017). Oleoresin  
409 residues remain in the final tocopherols product.

410  
411 **Evaluation Question #2: Discuss whether the petitioned substance is formulated or manufactured by a**  
412 **chemical process or created by naturally occurring biological processes [7 U.S.C. 6502(21)]. Discuss**  
413 **whether the petitioned substance is derived from an agricultural source.**

414  
415 *Synthetic/nonsynthetic classification*

416 As described in [Evaluation Question #1](#), formulation and chemical treatment of commercial food grade  
417 tocopherols are common ways to prevent degradation and increase shelf life. The free tocopherols may  
418 revert back into ester form in the presence of acetic or succinic acid (Moreau & Lampi, 2012). This chemical  
419 change is created by a synthetic process.

420  
421 Evaluation of tocopherols against Guidance NOP 5033-1 *Decision Tree for Classification of Materials as*  
422 *Synthetic or Nonsynthetic* (NOP, 2016a) is discussed below and summarized in [Table 3](#). Stabilization using  
423 acetic or succinic acid may occur in different manufacturing processes, but it is known to occur in solvent  
424 extraction. Therefore, we include discussion of this stabilization treatment under solvent extraction.  
425 However, this treatment may also be used on tocopherols from other manufacturing processes.

426  
427 Due to the potential use of antioxidants and other chemical treatments, we cannot categorically state  
428 whether some manufacturing processes will lead to a nonsynthetic product. For example, acetic/succinic  
429 acid treatment of any tocopherol (regardless of source) results in a synthetic product as this chemically  
430 transforms the tocopherols. Tocopherols are frequently extracted from nonagricultural sources, such as  
431 DDs, and often require a pretreatment step before extraction. Tocopherols sourced from deodorized  
432 distilled sludge (DD) are discussed together, below. All other sources are discussed by method of  
433 extraction instead of source. [Table 1](#) differentiates between these methods and sources.

#### 434 Tocopherols, from DD sources

435 DD sources are commercially used in supercritical fluid extraction (SFE), molecular distillation, matrix  
436 solid-phase dispersion, and some solvent extraction techniques (Soxhlet and alkaline-assisted solvent  
437 extraction). The evaluation below applies to extractions where those methods apply:

##### 438 1. *Is the substance manufactured, produced, or extracted from a natural source?*

439  
440 DDs are byproducts of the vegetable oils industry. The vegetable oils themselves are products sourced  
441 from a plant material. Oils are obtained from the original plant material by mechanical (e.g., pressing),  
442 enzymatic, or organic solvent (e.g., hexane) means. Synthetic soaps made during the separation process are  
443 removed. All other purification steps are physical and do not chemically change the tocopherols found in  
444 the oil source. Therefore, the answer to this question is yes, tocopherols are extracted from a natural source  
445 (DD, produced from vegetable oils).

##### 446 2b. *At the end of the extraction process, does the substance meet all of the criteria described at 4.6 of NOP 5033?*

447  
448 In order to separate tocopherols from the DD source material, DDs are first pretreated with physical and  
449 chemical methods (Liu et al., 2019). This pretreatment causes the esterification of free fatty acids with an  
450 organic solvent such as methanol and an acid catalyst (da Silva et al., 2023). Unreacted solvent is removed  
451 via vacuum distillation and the acid catalyst is removed by washing with water. The fatty acid product, an  
452 alkyl ester, is removed at a later step. Pretreatment of the DD material does not cause chemical changes in  
453 the free tocopherols. However, pretreatment can create impurities during the esterification process  
454 (tocopherol esters and quinones), which are synthetic. Free alpha-tocopherol is not completely isolated  
455 from these synthetic impurities by the end of the extraction process.

456  
457  
458 While the free alpha-tocopherol that survives the extraction process is not chemically changed, impurities  
459 created in the pretreatment process are synthetic and can remain.

---

<sup>8</sup> Oleoresin: a semi-solid extract composed of a resin component and a fatty acid component.

460

461 Tocopherols from non-DD sources462 1. *Is the substance manufactured, produced, or extracted from a natural source?*463 Yes. The substance, tocopherols, are extracted from natural sources such as seeds, leaves, grain, and other  
464 plant material or derivatives of these sources (i.e., oils). For the sake of simplicity, the steps taken to  
465 produce the derivatives are assumed to be nonsynthetic.

466

467 Tocopherols, produced from solvent extraction methods (direct solvent, alkaline-assisted solvent, PLE)468 2b. *At the end of the extraction process, does the substance meet all of the criteria described at 4.6 of NOP 5033?*

469

470 Solvent extraction and pressure liquid extraction (PLE)471 Yes. At the end of the solvent extraction processes, tocopherols remain largely as tocopherols without  
472 transformation of the compounds. The synthetic solvents used during the extraction are removed via  
473 evaporation. Solvent residues, present at impurity levels, do not have a functional effect. Due to the heat of  
474 evaporation, an impurity level amount of oleoresin transformation may occur. This impurity remains in the  
475 final tocopherols product; however, it is the result heating and not a synthetic chemical change per  
476 NOP 5033-1 (NOP, 2016a). This reasoning applies to extractions that do not require alkaline hydrolysis or  
477 conversion into the more stable ester form.

478

479 Similar to non-solvent extraction methods, we did not find any information regarding post-extraction  
480 steps. Antioxidants or acetic/succinic acid treatment may occur. Unless antioxidants or other undisclosed  
481 treatments are used, then tocopherols produced by PLE are nonsynthetic.

482

483 Alkaline-assisted solvent extraction (saponification)

484 No, at the end of the process, the substance does not meet all of the criteria described at 4.6 of NOP 5033.

485

486 Saponification, when employed, liberates tocopherols from the carbohydrates and proteins found in the  
487 source material. This process converts tocopherol esters into a different "free form" to obtain an extract  
488 with higher tocopherol content (Shammugasamy et al., 2013). At the same time that this process produces  
489 free tocopherols, it also unintentionally esterifies 4-5% of the total free tocols (tocopherols and tocotrienols)  
490 in the source material and these remain in the final tocopherols extract (Moreau & Lampi, 2012). The  
491 conversions of tocopherol esters to free tocopherols and the free tocols into tocol esters are reactions caused  
492 by the addition of the synthetic alkaline component.

493

494 Addition of antioxidants

495 No, at the end of the process, the substance does not meet all of the criteria described at 4.6 of NOP 5033.

496 Regardless of solvent extraction method, use of antioxidants to slow tocopherols' degradation is a common  
497 practice. These antioxidants (ascorbic acid, BHT, pyrogallol) are synthetic themselves.

498

499 Thus, the answer to whether tocopherols produced from the solvent extraction process meets all the criteria  
500 described at 4.6 of NOP 5033 in this case would be no, and the end product is considered synthetic. This  
501 evaluation would also apply to any other manufacturing methods using antioxidants.

502

503 Tocopherols, produced from ultrasonic-assisted extraction (UAE)504 2b. *At the end of the extraction process, does the substance meet all of the criteria described at 4.6 of NOP 5033?*505 Yes. Organic solvent "cuts" the plant material and releases the tocopherols present without transforming  
506 the compound. A small amount of tocopherols may degrade as a result of the cavitation bubble implosion  
507 heat.

508

509 The next question for tocopherols, produced from UAE is:

510 1. *Has the substance undergone a chemical change so that it is chemically different than how it naturally occurs  
511 in the source material?*

512 No, a chemical change does not occur. Therefore, the extraction of tocopherols by UAE is nonsynthetic.

513

514 Tocopherols, produced from matrix solid-phase dispersion (MSPD)

515 2b. *At the end of the extraction process, does the substance meet all of the criteria described at 4.6 of NOP 5033?*  
 516 Two main solvents are used in the MSPD method. The first solvent is used to suspend and aid in the  
 517 adsorption of the tocopherols onto the surface of the inert porous column material. Tocopherols remain  
 518 unchanged in this process. The adsorption process works by manipulating the polarity of the materials.  
 519 The second solvent detaches the tocopherols from the alumina. In the case of palm fatty acid distillate, the  
 520 third solvent used is a protic polar solvent, which cleans up any residual tocopherols that may remain after  
 521 the nonpolar hexane elution. At the end of the extraction process, tocopherols remain largely as  
 522 tocopherols without transformation of the compounds.

523  
 524 The next question for tocopherols, produced from MSPD is:

525 2. *Has the substance undergone a chemical change so that it is chemically different than how it naturally occurs*  
 526 *in the source material?*

527 No, a chemical change does not occur. Therefore, the extraction of tocopherols by MSPD is nonsynthetic.

528  
 529

**Table 3: Synthetic/nonsynthetic summary of methods**

| Extraction method or source                               | Agricultural source? | Chemical change?                                  | Other materials present? | Synthetic/Nonsynthetic                      |
|---|----------------------|---|--------------------------|---|
| DD source (including molecular distillation)              | Yes                  | No (excluding impurities) <sup>9</sup>            | Yes, impurities          | Nonsynthetic, but with synthetic impurities |
| Solvent extraction (including Soxhlet, alkaline-assisted) | Yes                  | Yes, due to reaction with succinic or acetic acid | Yes, additives           | Synthetic                                   |
| UAE   | Yes                  | No  | No                       | Nonsynthetic                                |
| MSPD  | Yes                  | No  | No                       | Nonsynthetic                                |

530

531 Agricultural/nonagricultural classification

532 Evaluation of tocopherols against Guidance NOP 5033-2 *Decision Tree for Classification of Agricultural and*  
 533 *Nonagricultural Materials for Organic Livestock Production or Handling* (NOP, 2016b) is discussed below.

534

535 DD sources

536 1. *Is the substance a mineral or bacterial culture, as included in the definition of nonagricultural substances at*  
 537 *section 205.2 of the USDA organic regulations?*

538 No. DDs are substances that are a fraction of crude plant oil.

539

540 2. *Is the substance a microorganism (e.g., yeast, bacteria, fungi) or enzyme?*

541 No. It is a plant oil fraction.

542

543 3. *Is the substance a crop or livestock product or derived from crops or livestock?*

544 Yes. DDs are the inedible fraction of crude oil derived from crops (e.g. corn, soybean, etc.).

545

546 4. *Has the substance been processed to the extent that its chemical structure has been changed?*

547 Yes, but only a portion of the material (impurities) is chemically changed. Chemical pretreatment and  
 548 subsequent temperature changes causes a portion of the tocopherols to form synthetic substances  
 549 (tocopherol esters and tocopherol quinones), which themselves do not exist in the plant tissue the  
 550 tocopherols were isolated from. Tocopherol esters and quinones are also created in the pretreatment step  
 551 using methanol or ethanol and are indistinguishable from those created from heat degradation.

552

<sup>9</sup> Tocopherol esters and tocopherol quinones are formed as impurities in the DD manufacturing process. It is possible that these are formed in other manufacturing processes that use high temperature processing steps as well. However, this is not discussed in literature. These impurities are synthetic and remain in the final free alpha-tocopherol product. These are formed as impurities, and they may or may not have a functional effect in the final product. These impurities are impossible to distinguish from the free alpha-tocopherols because of their similar physicochemical properties. NOP 5033-1 (NOP, 2016a) does not provide guidance on whether to include impurities within the classification of materials.

553 The desired end product, free alpha-tocopherols, are not chemically changed (and therefore are classified  
554 as agricultural).

555

556 5. *Is the chemical change a result of naturally occurring biological processes such as fermentation or use of*  
557 *enzymes; or a result of mechanical/physical/biological process described under section 205.270(a)?*

558 Applies to the impurities only: No. Transformation through heat is described as a  
559 mechanical/physical/biological process under § 205.270(a). However, an undetermined, indistinguishable  
560 amount of the same impurities (tocopherol esters and tocopherol quinones) are a result of a chemical  
561 change through the esterification process that isolates fatty acids from the rest of the DD.

562

563 The impurities are therefore classified as nonagricultural.

564

565 All other sources

566 1. *Is the substance a mineral or bacterial culture, as included in the definition of nonagricultural substances at*  
567 *section 205.2 of the USDA organic regulations?*

568 No. Tocopherols are a series of compounds, isolated from plant sources.

569

570 2. *Is the substance a microorganism (e.g., yeast, bacteria, fungi) or enzyme?*

571 No. Tocopherols are compounds isolated from plant sources.

572

573 3. *Is the substance a crop or livestock product or derived from crops or livestock?*

574 Yes. Tocopherols are derived from plant material.

575

576 4. *Has the substance been processed to the extent that its chemical structure has been changed?*

577 Applies to methods where additives are not used: No. The structure of tocopherols remains unchanged.  
578 However, these isolates may degrade over time into their oxidized forms. Therefore, tocopherols are an  
579 agricultural material, according to the decision tree.

580

581 Applies to methods where reactants and antioxidants are used: Yes. Tocopherols are reacted with additives  
582 to improve their stability.

583

584 5. *Is the chemical change a result of naturally occurring biological processes such as fermentation or use of*  
585 *enzymes; or a result of mechanical/physical/biological process described under section 205.270(a)?*

586 No. Generally, unbound tocopherols are reacted with acetic or succinic acid to improve their stability. This  
587 reaction yields a nonagricultural product. Also, nonagricultural additives in the form of BHT, pyrogallol,  
588 and ascorbic acid may be used and included in the identity of the final tocopherols product. These  
589 compounds physiochemically interact with the tocopherols to avoid degradation but do not form new,  
590 permanent compounds.

591

592 The literature does not specify if these treatments extend to less commonly used methods of extraction  
593 (PLE, MSPD, and UAE). PLE, MSPD, and UAE methods should be evaluated for additives and stabilizing  
594 agents. In cases where additives are used, the agricultural/nonagricultural determination may depend on  
595 the ancillary ingredient policy of a given accredited certification agency.

596

597 **Evaluation Question #3: If the substance is a synthetic substance, provide a list of nonsynthetic or**  
598 **natural source(s) of the petitioned substance [7 CFR 205.600(b)(1)].**

599

600 *Nonsynthetic sources with tocopherols*

601 Many nonsynthetic substances contain high quantities of tocopherols. However, these sources are not  
602 purified isolates, and contain other compounds. For example, wheat germ oil is rich in tocopherols  
603 (Moreau & Lampi, 2012). Vitamin E and tocopherols were discovered in wheat germ oil. However, we did  
604 not find literature describing the use of oils such as this as an antioxidant additive, or as a replacement for  
605 refined tocopherols.

606

607 *Availability of nonsynthetic tocopherols*

608 There is at least one certified organic tocopherol product in the market. Currently, the Organic Integrity  
 609 Database (2023) shows that there is one vitamin E product marketed as “D-alpha tocopherol.” Other  
 610 certified organic vitamin E products are available; however, these do not specify which form of vitamin E is  
 611 included in that product. Although tocopherols include vitamin E products, not all vitamin E products  
 612 include tocopherols. Instead, vitamin E is a generic term for substances that have the same activity as a  
 613 stereoisomer of one type of tocopherol (Sen et al., 2006).<sup>10</sup> Tocotrienols, or a combination of tocotrienols  
 614 and tocopherols, may also be described with this term (Rich et al., 2012; Sen et al., 2006).

615  
 616 **Evaluation Question #13: Provide a list of organic agricultural products that could be alternatives for the  
 617 petitioned substance [7 CFR 205.600(b)(1)].**

618 Tocopherols (alpha-, beta-, gamma-, and delta-tocopherol) are used to prevent lipid oxidation, a problem in  
 619 foods that produces rancid odors and flavors, decreases shelf life, alters texture and color, and decreases  
 620 nutritional value (Alamed et al., 2009). Therefore, alternatives to tocopherols should prevent lipid  
 621 oxidation. The material allowance at § 205.605(b)(36) mentions that tocopherols may be used when  
 622 rosemary extracts are not a suitable alternative.

623  
 624 There is at least one certified organic tocopherols product in the market, marketed as “D-alpha tocopherol”  
 625 (see [Evaluation Question #3](#)).

626  
627 *Alternatives*

628 Most commercial food antioxidants work by scavenging free radicals or chelating metals (Alamed et al.,  
 629 2009). There are at least 26 spices, 51 edible and wild flowers, and 223 medicinal plants showing  
 630 antioxidant activity (Xu et al., 2017). One alternative example is rosemary extract, which is authorized in  
 631 the EU for an antioxidant use (Coulombier et al., 2021; Karre et al., 2013). Another alternative is green tea  
 632 extracts, which contain a mixture of several compounds known to inhibit lipid peroxidation (Yin et al.,  
 633 2012).

634  
 635 **Table 4. Spice extracts and compounds of interest for use as alternates to prevent lipid oxidation. Sources: Li et al.,  
 636 2014; Shan et al., 2005; Yin et al., 2012.**

| Botanical extract | Scientific name               | Antioxidant compound | Concentration (mg/100 g of dry weight) |
|-------------------|-------------------------------|----------------------|--|
| Mint              | <i>Mentha canadensis</i>      | Rosmarinic acid      | 1908.5                                 |
| Sweet basil       | <i>Ocimum basilicum</i>       | Coumaric acid        | 27.9                                   |
|                   |                               | Rosmarinic acid      | 1086.1                                 |
| Oregano           | <i>Origanum vulgare</i>       | Coumaric acid        | 214.8                                  |
|                   |                               | Rosmarinic acid      | 2562.7                                 |
| Rosemary          | <i>Rosmarinus officinalis</i> | Rosmarinic acid      | 1286.4                                 |
| Sage              | <i>Salvia officinalis</i>     | Coumaric acid        | 40.0                                   |
|                   |                               | Rosmarinic acid      | 2186.1                                 |
| Thyme             | <i>Thymus vulgaris</i>        | Gallic acid          | 37.5                                   |
|                   |                               | Coumaric acid        | 55.9                                   |
|                   |                               | Rosmarinic acid      | 681.1                                  |
| Clove             | <i>Eugenia caryophyllata</i>  | Gallic acid          | 783.5                                  |
| Sea lavender      | <i>Limonium sinautum</i>      | Catechins            | 1711.2                                 |
| Garden geranium   | <i>Pelargonium hortorum</i>   | Catechin             | 693.1                                  |
| Tea olive         | <i>Osmanthus fragrans</i>     | Catechins            | 1946.4                                 |
| Green tea         |                               | Catechins            | ---                                    |

637  
638 *Performance and test data*

639 Alamed et. al. (2009) compared different antioxidant activities in an oil-in-water emulsion of beef. The  
 640 study compared ferulic acid, coumaric acid, propyl gallate, gallic acid, rosmarinic acid, ascorbic acid, and  
 641 alpha-tocopherol. Propyl gallate, gallic acid, and rosmarinic acid showed promising free radical scavenging  
 642 properties. However, the results were inconclusive because of the variety of factors (processing conditions,  
 643 fatty acid composition, product matrix, etc.) that can affect the ability of a compound to inhibit lipid  
 644 oxidation.

<sup>10</sup> Stereoisomer: A compound that differs only in spatial orientation of the atoms. The chemical formula is the same.



645  
646 Of the above compounds, rosmarinic, gallic, and coumaric acid are extracts of botanical substances. Shan et  
647 al. (2005) analyzed an assortment of herbs in the *Labiatae* family for their phenolic compound content (see  
648 [Table 2](#)). The researchers noted that the herbs with the highest antioxidant capacity were clove, cinnamon,  
649 and oregano. Mint, sweet basil, oregano, and rosemary were also studied (Shan et al., 2005). Rosmarinic  
650 acid was the highest-concentration phenolic compound in the six species analyzed.

651  
652 Alizadeh et al. (2016) compared the antioxidant activities of tocopherols, rosemary extract, ferulago, and a  
653 synthetic antioxidant (TBHQ) on a sunflower seed oil and palm olein mixture (1:1) during the deep frying  
654 of potato slices. The study noted that tocopherols were effective in controlling the primary stage oxidation  
655 while rosemary extract inhibited both primary and secondary oxidation, more akin to the synthetic THBQ.  
656 Ferulago showed secondary oxidation inhibitive effects only.

657  
658 An additional study by Karre et al. (2013) reported on rosemary, oregano, thyme, and sage extracts in the  
659 context of packaged meat products. The study noted that rosemary is effective in turkey meat, raw ground  
660 beef and pork, cooked ground beef, and raw frozen sausage. Oregano, though effective, is less effective  
661 than sage and thyme extracts.

662  
663 As a part of a synergism study, Yin et al. (2012) reported on the effects of green tea extract as a food  
664 additive when compared to alpha-tocopherol in vegetable oils. Green tea extract was more efficient than  
665 alpha-tocopherol as an antioxidant in pure bulk sunflower oil. The paper briefly discussed other studies  
666 that attribute this to hydrophilic antioxidants being more efficient in lard systems. The researchers also  
667 noted that some other studies have found that hydrophobic antioxidants are more effective than  
668 hydrophilic ones. The researchers also found that alpha-tocopherol showed higher antioxidant activity  
669 than green tea extract when in a methyl linoleate emulsion.

670  
671 *Commercial availability*

672 Certified organic botanical extracts are easily available in the market. All sources provided on [Table 4](#) have  
673 certified organic sources available (USDA, 2023).

674

## Report Authorship

675

676 The following individuals were involved in research, data collection, writing, editing, and/or final  
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682

683 All individuals are in compliance with Federal Acquisition Regulations (FAR) Subpart 3.11 – Preventing  
684 Personal Conflicts of Interest for Contractor Employees Performing Acquisition Functions.

685

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