# Properties and Characteristics of Amine and Ester Formulations of 2,4-D

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- 2,4-D is generally formulated as an amine salt or an ester, each of which have their own advantages and tradeoffs.
- Generally, ester formulations are considered more efficacious, but more likely to drift off target, while amine salts are considered less efficacious but more stable.
- Some phenoxy herbicides can be used interchangeably, but others cannot.

## Introduction

2,4-D is a post-emergence systemic herbicide used widely for selective control of broadleaf weeds in a variety of sectors and is found in many commonly used products. Currently, there are over fifty 2,4-D products labeled in the United States and a similar number of products that contain 2,4-D in combination with other herbicides (Peterson et al, 2016). There are over 600 combination products if one includes turf weed and feed products.

In its pure form, 2,4-D acid is a dry crystalline solid and must be formulated to readily disperse and form a suitable mixture with water. To accomplish this, 2,4-D is produced in several forms, including acids, salts, amines and esters. Amine salts and esters are the two most common formulation types that have gained widespread acceptance in the marketplace. Of these, the dimethylamine salt (DMA) and ethylhexyl ester (EHE) forms account for approximately 90-95% of the total global use (Charles et al, 2001). Amines and esters are different in many respects, and each offers advantages and disadvantages for certain uses. Some general differences in amine and ester formulations include the following:

- Esters are absorbed more quickly than amines on broadleaf weeds and are more efficient under certain environmental conditions and for the control of certain plant species.
- Amine formulations of 2,4-D are essentially non-volatile, and pose less potential for vapor movement following application.
- Esters are absorbed more quickly by plants and therefore are less likely to be washed away by rain.
- Amines are often thought of as being less phytotoxic to crops than ester formulations, however, this is not accurate for all crops and all situations.

This document details the main characteristics of 2,4-D amine and ester formulations to help explain these and other differences. 2,4-D formulation differences are widely recognized in the industry and among users, and products that contain each formulation are clearly positioned to maximize efficacy and minimize risk.

## Amine and Ester Formulations of 2,4-D

*(The following formulation descriptions are adapted from Peterson, M.A., S.A. McMaster, D.E. Riechers, J. Skelton, and P.W. Stahlman. 2016. 2,4-D Past, Present, and Future: A Review. Weed Technology 30:303–345.)*

### Amine Salts

When the acid of 2,4-D is reacted with an amine, the salt of 2,4-D is formed. The salt-based formulation renders the 2,4-D acid active ingredient water soluble. Common amine salt formulations of 2,4-D include isopropyl amine, triisopropanolamine, diethanolamine, and dimethylamine. The latter is the most widely used. Amine formulations are readily soluble in water (greater than 50% by weight) and form a true solution. The amine salts have gradually replaced mineral salts (lithium, potassium, and sodium) because the amine salts are more readily dissolved in water. Recently a choline salt of 2,4-D has been developed by Dow AgroSciences with greater stability and lower opportunity for volatilization than other amine forms of 2,4-D.

When placed in water, these salt formulations dissociate, or separate, into the acid anion, , which carries a negative charge, and the amine cation, which carries a positive charge. In hard water, which is high in calcium and magnesium ions (Ca2+ and Mg2+), these and other cations can associate with the negatively charged 2,4-D acid anion and form insoluble salts that can precipitate, reducing the amount of herbicide in solution and plugging line screens and nozzle body screens on a sprayer. For this reason, sequestering agents are included in most amine formulations to reduce or eliminate this problem. Most amine salts of 2,4-D form a clear solution when dissolved in water; however, the addition of sequestering agents to the formulation can impart a darker, amber color. This change in color has no impact on the biological activity or crop tolerance of the formulation. Most amine salts are not soluble in petroleum oils. Salts can be formulated as liquid or dry preparations.

### Esters

Reaction of 2,4-D acid with an alcohol forms a 2,4-D ester. Some ester forms of 2,4-D include butoxyethyl ester (BEE), 2-ethylhexyl ester (2-EHE, previously known as iso-octyl ester), ethyl ester, propylene glycol butyl ether ester, methyl ester, isopropyl ester, and butyl ester. BEE and 2-EHE have gradually replaced the other esters mentioned and are the only technical esters being supported for US EPA reregistration, with the exception of special use for the isopropyl ester on citrus. Esters made from alcohols with an alkyl chain of four carbons or fewer are considered more volatile. This group includes the methyl, isopropyl, and butyl esters. Removal of these formulations for most uses from the commercial channels in the early 1980s significantly reduced off-target injury to sensitive plants. On the other hand, esters of 2,4-D made from alcohols with an alkyl chain of more than four carbons are classified as low-volatile esters. It is important to remember that the longer the carbon chain the lower the volatility. The 2,4-D esters made from long-chain alcohols that are classified as low-volatile esters include BEE and 2-EHE (which has a chain of eight carbons).

Esters are readily soluble in petroleum oils but are insoluble in water. For this reason they are formulated as emulsifiable concentrates for application in either water or oils. When an emulsifiable concentrate formulation of 2,4-D ester is mixed with water, the emulsifying agent keeps the tiny oil-like droplets suspended and dispersed, and the ester is held in an oil-in-water type of emulsion. The 2,4-D emulsion appears milky. If allowed to stand, the oil droplets may separate; however, mixing will reform the emulsion.

## Other Formulations

Over the years, many different formulations of 2,4-D have been produced. Acid formulations, both dry and liquid, have been marketed, as well as salts such as lithium, sodium, trolamine, and others. These formulations have seen limited use and in some cases are no longer commercially available.

The physical and chemical properties of the major 2,4-D formulations are presented in Table 1, on the following page.

Table 1. Physical–Chemical Properties of 2,4-D and Associated Formulations.

| Active Ingredient | Form | Vapor Pressure* | Molecular Weight | Solubility in Water (mg/L)** | Log $K_{ow}$ |
|---|---|---|---|---|---|
| 2,4-D acid | White to brown crystalline solid | $1.9 \times 10^{-5}$ Pa<br>$1.4 \times 10^{-7}$ mmHg | 221 | pH 5: 29,934 ± 2957<br>pH 7: 44,558 ± 674<br>pH 9: 43,134 ± 336 | 0.001 M sol'n<br>pH 5: 2.14<br>pH 7: 0.177<br>pH 9: 0.102 |
| 2,4-D Sodium salt | White powder | Salt dissociates to acid in water | 243.03 | 45,000 mg/L | Salt dissociates to acid in water |
| 2,4-D-diethanolamine salt (DEA) | Cream colored powder | $9.98 \times 10^{-8}$ mmHg | 326.18 | 806,000 mg/L | $2.24 \times 10^{-2}$<br>-1.65 |
| 2,4-D dimethylamine (DMA) salt | Amber aqueous liquid | $1.33 \times 10^{-5}$ Pa<br>$1 \times 10^{-7}$ mmHg | 266.13 | pH 5: 320,632 ± 3645<br>pH 7: 729,397 ± 86,400<br>pH 9: 663,755 ± 94,647 | See values for 2,4-D acid above |
| 2,4-D - isopropylamine (IPA) salt | Amber aqueous liquid | Salt dissociates to acid in water | 280.04 | pH 5: 174,000 mg/L<br>pH 7: 436,000 mg/L<br>pH 9: 331,000 mg/L | Salt dissociates to acid in water |
| 2,4-D tri-isopropanolamine (TIPA) salt | Amber aqueous liquid | Salt dissociates to acid in water | 412.31 | pH 5: 461,000 mg/L<br>pH 7: 461,000 mg/L<br>pH 9: 104,000 mg/L | Salt dissociates to acid in water |
| 2,4-D-choline salt | Amber aqueous liquid | Salt dissociates to acid in water | 324.7 | 777,000 mg/L | Salt dissociates to acid in water |
| 2,4-D butoxyethyl ester (BEE) | Dark amber liquid | $3.2 \times 10^{-4}$ Pa<br>$2.4 \times 10^{-6}$ mmHg | 321.2 | Practically insoluble in water | 4.1 |
| 2,4-D 2-ethylhexyl ester (EHE) | Dark amber liquid | $4.8 \times 10^{-4}$ Pa<br>$3.6 \times 10^{-6}$ mmHg | 333.27 | 0.0867 mg/L | 5.78 |
| 2,4-D -isopropyl ester (IPE) | Pale amber liquid | 1.87 Pa<br>$5.3 \times 10^{-6}$ mbar | 263.12 | Practically insoluble in water | 253.8 ± 44.4 |

Source: 2,4-D Technical fact sheet. National Pesticide Information Center. http://npic.orst.edu/factsheets/archive/2,4-DTech.html. Accessed August 8, 2016.
*Vapor pressure measured at 25 °C.
**Solubility in water given for unbuffered solution.

## Differential Characteristics of Amine and Ester Formulations

### Uptake and Translocation

As described in the previous section, the ester forms of 2,4-D are readily soluble in petroleum oils, but are insoluble in water, while amine forms are water soluble. Ester formulations, due to their oil based nature, penetrate the waxy skin (cuticle) of the plant leaf surface more effectively than amine forms (Rao, 2000). In general, ester forms of 2,4-D can have greater biological activity than amine salt formulations under some circumstances, such as early in the season when temperatures are cool or fluctuating or under dry conditions when plants are not growing as actively, or for control of plant species that have a thick, waxy, difficult to penetrate cuticle (Praczyk et al 2012). Esters may, however, cause greater injury on some crops because of faster uptake, which may temporarily overwhelm the plants' detoxification mechanisms. Ester formulations are more rain resistant than salt formulations because they quickly penetrate the cuticle. The rainfast interval for 2,4-D amine is 4-8 hours compared to 1 hour for 2,4-D ester. Once absorbed into the plant, both the amine and ester are converted to the acid and accumulate in cells passively, or are actively transported within the plant, accumulating at root and shoot growth areas.

### Soil Activity

2,4-D has a relatively short soil half-life of approximately 6 days (Peterson et al., 2016). Amine and ester formulations are rapidly converted to the acid form once they contact soil, and the rate of dissipation from soils is often the same regardless of the formulation of 2,4-D applied (Tu et al., 2001). Label restrictions regarding the interval between burndown applications and planting of soybeans vary by formulation, with 2,4-D ester having a shorter interval than 2,4-D amine. Ester rates up to 0.5 lb ae/A must be applied at least 7 days before planting, and rates between 0.5 and 1 lb ae/A must be applied at least 15 days before planting. This compares to amine rates up to 0.5 lb ae/A which must be applied at least 15 days before planting, and rates between 0.5 and 1 lb ae/A which must be applied at least 30 days before planting.

### Drift and Volatility

Off-target movement of herbicides can occur through physical drift of the liquid spray solution, or through volatilization and subsequent vapor drift of the chemical. Spray drift is the downwind movement of spray particles from the application site to non-target areas. All herbicides are susceptible to off-target movement through physical drift of the liquid spray solution, and most crop injury is caused by spray drift. The extent of spray drift increases as the size of spray droplets decreases, the height above the ground from which the droplets are released increases, and wind speed increases.

Vapor movement of herbicides occurs when molecules of the active ingredient convert from a liquid to a gas and are transported off the application site by wind or air currents. Vaporization is a function of both the inherent physical properties of the herbicide and the environmental conditions present during and after the application. One of the commonly used methods to measure a substance's volatility is its vapor pressure. The higher the vapor pressure of a substance, the greater its tendency to volatilize, although this is not the only factor influencing volatility. Vapor pressures for 2,4-D formulations, other substances, and commonly used herbicides are presented in Tables 2 and 3. Although 2,4-D ester formulations are relatively more volatile than 2,4-D acid and amine formulations, all forms of 2,4-D have low vapor pressures relative to many other substances and commonly used herbicides.

Table 2. Vapor Pressures for 2,4-D Formulations and Other Common Substances.

| Substance | Vapor Pressure (mmHg) | Temperature |
|---|---|---|
| 2,4-D DMA | $<1.0 \times 10^{-7}$ | 25 C |
| 2,4-D acid | $1.4 \times 10^{-7}$ | 25 C |
| 2,4-D BEE | $2.4 \times 10^{-6}$ | 25 C |
| 2,4-D EHE | $3.6 \times 10^{-6}$ | 25 C |
| Ethylene glycol | 3.75 | 20 C |
| Water ($H_2O$) | 17.5 | 20 C |
| Propanol | 18.0 | 20 C |
| Ethanol | 43.7 | 20 C |
| Freon 113 | 284 | 20 C |
| Butane | 1,650 | 20 C |
| Propane | 7,584 | 26.85 C |
| Carbon dioxide | 42,753 | 20 C |

Abbreviations: DMA = dimethylamine, BEE = butoxyethyl ester, EHE = 2-ethylhexyl ester

Table 3. Vapor Pressures for 2,4-D Formulations and Several Commonly Used Herbicides.

| Herbicide | Vapor Pressure (mmHg) | Temperature |
|---|---|---|
| Glyphosate IPA | $1.58 \times 10^{-8}$ | 25 C |
| Glyphosate Ammonium Salt | $6.75 \times 10^{-8}$ | 25 C |
| 2,4-D DMA | $<1.0 \times 10^{-7}$ | 25 C |
| 2,4-D Acid | $1.4 \times 10^{-7}$ | 25 C |
| Atrazine | $2.9 \times 10^{-7}$ | 25 C |
| 2,4-D BEE | $2.4 \times 10^{-6}$ | 25 C |
| 2,4-D EHE | $3.6 \times 10^{-6}$ | 25 C |
| Trifluralin | $1.1 \times 10^{-4}$ | 25 C |
| Clomazone | $1.4 \times 10^{-4}$ | 25 C |
| EPTC | $3.4 \times 10^{-2}$ | 25 C |

Abbreviations: IPA = isopropylamine, DMA = dimethylamine, BEE = butoxyethyl ester, EHE = 2-ethylhexyl ester, For most herbicides, volatility is of little consequence regardless of vapor pressure because of the herbicide's mode of action, and the symptoms which develop on exposed plants. EPTC, for example, is so volatile that it must be incorporated into the soil to prevent drift, however, if it does drift, it does not injure surrounding plants. Clomazone, on the other hand, possesses a vapor pressure approximately 240 times lower than EPTC and which is just barely above the requirement for incorporation of 1.0 x 10-4mm Hg. However, because of the distinctive symptoms clomazone produces, spray and vapor off-target movement can result in highly visible injury symptoms in surrounding fields. For this reason, a micro-encapsulated formulation was developed, and is now marketed to help prevent vapor loss. 2,4-D EHE possesses a vapor pressure nearly 10,000 times lower than EPTC, however, because of 2,4-D's mode of action and the sensitivity of certain plant species, plants exposed to very low concentrations of 2,4-D display diagnostic symptoms. Amine forms of 2,4-D have lower vapor pressures than esters, and once both the amine and ester forms convert to the acid, vaporization generally approximates that of the acid.

## Effect of Temperature

It is commonly known that correct timing is crucial for acceptable weed control. This also holds true when deciding what formulation to use; ester or amine salt. During cooler weather (early spring and late fall), ester formulations can be used safely, and typically provide better weed control than amines. Volatility losses are low at cooler temperatures, and sensitive vegetation either has not yet emerged or is less sensitive to exposure at this time. As temperature increases, the risk of ester volatilization increases, and a switch to amine formulations is recommended in most situations. In warmer temperatures, amines and esters provide nearly equal weed control (Tanner, 2014). Also, some esters can be phytotoxic, and can injure crops and turfgrass when temperatures are too warm. Many herbicide labels list a temperature range in which they should be used, and these recommendations should be followed.

2,4-D formulation differences are widely recognized in the industry and among users. Products that contain each formulation are clearly positioned to maximize the benefits and minimize the risks associated with use. For example, professional turfgrass managers follow a three season approach to optimize weed control performance, and minimize off-target movement risk. An ester is used in the spring of the year for improved early season weed control. When maximum daytime temperatures reach 75 to 80 F, a switch is made to amine formulation in order to minimize off-target movement. Once maximum daytime temperatures fall below 65 F in the fall, a switch is made back to the ester as plants become more difficult to control and off-target movement risks diminish.

Preplant herbicide applications in corn and soybeans offer another example of how the industry has adopted product stewardship practices to maximize the benefits and minimize the risks of each 2,4-D formulation. Preplant burndown herbicide applications are typically made early in the season when weed control conditions are difficult and off-target movement presents little risk. Ohio State and Purdue University recommend the use of 2,4-D low-volatile ester for preplant use in corn and soybeans because of improved performance on difficult-to-control weeds such as glyphosate resistant marestail (horseweed), and the risk of crop injury from preplant applied amine and acid formulations. However, the 2016 Ohio, Indiana, Illinois Weed Control Guide still cautions that:

> *"Dicamba and the ester formulations of 2,4-D may vaporize at temperatures as low as 70 F and move with prevailing air currents to areas with sensitive plants, including ornamentals, soybeans, and vegetable crops. Amine formulations of 2,4-D are essentially nonvolatile. ….The rate of herbicide volatilization increases with increasing temperature. In the summer, temperatures at the soil surface may exceed 140°F on a clear day, greatly enhancing conditions for volatility. Vapors drift farther and over a longer period of time than do spray droplets. Changes in temperature and wind direction following application can move damaging vapors to sensitive plants. To avoid vapor drift, carefully observe label precautions when applying a volatile herbicide."*

No one wants to be sued or anger a neighbor over off-target movement. The industry and users have adopted product stewardship practices which manage the risks effectively, and reap the unique benefits of each formulation. In addition, this symbiotic relationship between the amine and ester formulations may also reduce the pounds of herbicide active ingredients applied as well as the risk of crop injury. For example, higher rates of an amine would be required to compensate for lower activity when used early in the season. The use of higher rates for preplant burndown may increase the risk of crop injury. Alternatively, higher rates of amine would be required to control larger weeds if the application were delayed until warmer temperatures provided more favorable conditions for amine performance. Conversely, esters used later in the season would increase the risk of off-target movement and crop injury. The combined benefits of amine and ester formulations are critical to optimize the use of 2,4-D in Integrated Pest Management (IPM) and Integrated Resistance Management (IRM) programs.

## Spray Carrier Quality and Tankmix Antagonism

As discussed in previous sections, hard water cations or micronutrients such as calcium, magnesium, manganese, sodium, and iron reduce the activity of amine formulations. The degree of antagonism is determined by the salt concentration. At low salt levels, weed control may not decrease under normal environmental conditions, but will be more noticeable under drought conditions or when applied to partially susceptible weeds. Ester formulations do not dissociate or ionize when added to water like amine formulations, and do not react with cations in hard water to form insoluble precipitates (Peterson et al., 2016).

Potassium salt formulations of glyphosate may negatively interact with amine salt formulations of 2,4-D in the spray tank, resulting in precipitation. This is an example of two dissimilar salts causing physical incompatibility, and the possibility of reduced weed control. Factors that increase the risk of precipitation are low spray volume, cold water, pH, and high herbicide rates. Another example of negative herbicide salt interaction is antagonism from tank- mixing of some salts of glyphosate and 2,4-D, such as glyphosate IPA (isopropylamine) and 2,4-D DMA on certain grassy weed species.

In small grains, neither amine nor ester formulations of 2,4-D can be tank mixed with Axial®[i] Star/Axial XL, Foxfire™[ii], Huskie®[iii] Complete, Puma®[iv]/Parity™v, Olympus®[vi] Flex, Orion®[vii], Rimfire®[viii] Max, or Silverado ™[ix] because of antagonism, however, MCPA ester may be mixed with these herbicides.

## Premix Formulations

Prepackaged combinations of 2,4-D with one or more herbicides are becoming more common in all markets. While premixed products clearly have advantages, one issue to consider is the compatibility of the premix partners. Typically, amine formulations of 2,4-D are used to premix with amine formulation premix partners, and ester formulations of 2,4-D are used to premix with ester formulation premix partners because of the different solvent systems used to formulate the products. Certain premix combinations would not be possible or more difficult to formulate if both 2,4-D forms (amines and esters) were not available.

## Extent of Interchangeability Among Phenoxy Herbicides

*By William M. Mahlburg, RegVantage Strategies LLC*

Phenoxy herbicides comprise a group of synthetic chemicals structurally related to the plant growth hormone indoleacetic acid. They are agronomically and economically useful because of their characteristic tolerance by monocotyledon plants (e.g., grasses) and phytotoxicity to dicotyledon plants (e.g., broadleaf weeds) at relevant application rates. This selectivity provides great advantages for weed management in crops, turf and other noncrop sites.

Among the best known phenoxy herbicides registered for use in the USA today are 2,4-D, 2,4-DB, dichlorprop-p, MCPA, MCPB and mecoprop-p. These herbicides may be used singly, mixed with non-phenoxy herbicides in either premixed combinations or at the time of use, or in certain cases, mixed with each other in either premixed combinations or at the time of

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[i] Axial is a registered trademark of Syngenta Crop Protection, LLC.
[ii] Foxfire is a registered trademark of Syngenta Crop Protection, LLC.
[iii] Huskie a registered trademark of Bayer CropScience.
[iv] Puma a registered trademark of Bayer CropScience.
[v] Parity is a registered trademark of Tenkox, Inc.
[vi] Olympus a registered trademark of Bayer CropScience.
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use. For example, premixed combinations of 2,4-D and mecoprop-p are common because of their utility in broadening the spectrum of weeds controlled in turf weed management. In such mixtures, MCPA is sometimes used interchangeably with 2,4-D, albeit at a slightly higher rate for similar effect. Additional herbicides may be included for even broader control.

In woody plant and brush control, premixed combinations of 2,4-D and dichlorprop-p are utilized to augment the range and extent of control. Again, additional herbicides may be included for even broader control. It is also the case that several of the phenoxy herbicides are almost never combined or used interchangeably because their activity is rather different from one another. A notable example is 2,4-D versus 2,4-DB. Crops like peanuts, soybeans and seedling alfalfa tolerate 2,4-DB because the herbicide must first undergo beta-oxidation in the plant before it expresses its herbicidal effect. The named crops are unable to provide the needed beta-oxidation, and thus a margin of crop safety is provided versus weeds that are able to provide beta-oxidation. Therefore, 2,4-D cannot be substituted for or combined with 2,4-DB and retain the necessary margin of crop safety. This is why 2,4-D and 2,4-DB are generally registered for use on dissimilar crops.

This section is provided to characterize the extent of interchangeability among phenoxy herbicides in sufficient detail that typical uses of the herbicides can be understood by the reader. Unlikely interchangeabilities are also apparent or described in the accompanying table. Crop and weed selectivity vary appreciably among phenoxy herbicides, but due to the large volume of information, those details are not exhaustively presented here.

The largest number of registered uses among phenoxy herbicides are those of 2,4-D, which span scores of crops and noncrop sites. Not surprisingly, 2,4-D is also used in the largest quantity of any of the phenoxy herbicides.

## Conclusions

2,4-D is a postemergence systemic herbicide used widely for selective control of broadleaf plants in cultivated agriculture, pasture and rangeland, turfgrass, forests, rights-of-way, and industrial sites, and for the control of aquatic vegetation and invasive plants. 2,4-D is produced in several forms including acids, salts, amines, and esters to readily disperse and form a suitable mixture with water. There are two main types of formulations, amine salts and esters, which have gained widespread acceptance in the marketplace. Of these, the DMA salt and EHE ester forms account for approximately 90-95% of the total global use. Amines are different to esters in many respects, and each offers advantages and disadvantage for certain uses. Depending on the time of year, location, weed pressure, and application technique, both forms can safely be used to achieve acceptable weed control. 2,4-D formulation differences are widely recognized in the industry and among users. The industry and users have adopted product stewardship practices which manage the risks effectively and reap the unique benefits of each formulation. Better product knowledge and application technologies have each played an important role in improving the stewardship of 2,4-D products, and new technological advancements will continue to improve its performance and safety benefits. Both amine and ester formulations are equally important weed management tools which benefit society by helping to ensure an abundant, healthy, low cost food supply, and a healthy and productive environment for the American public.

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