

COMPOSITION

The present invention relates to salts of ethylenediamine disuccinic acid (or EDDS). It also relates to methods of preparing such salts, uses thereof and compositions containing said salts. In particular the present invention relates to magnesium-containing salts of EDDS.

Ethylenediamine disuccinic acid has the structure shown in figure 1:

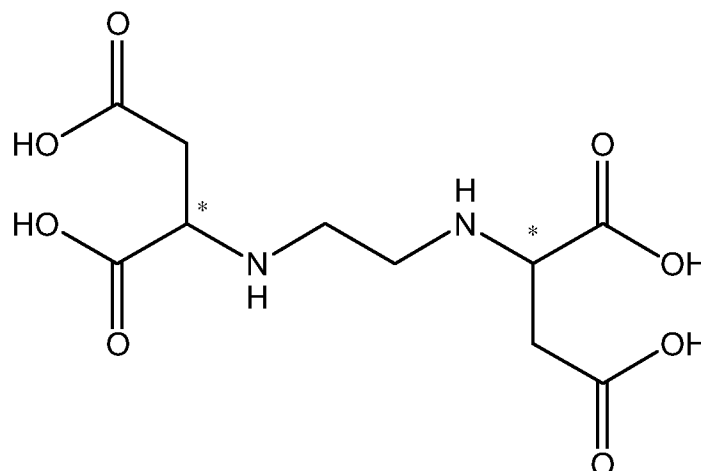


Figure 1

The structure includes two stereogenic centres and three possible stereoisomers exist. An especially preferred configuration is S,S ethylenediamine disuccinic acid as this compound is readily biodegradable.

Compositions comprising ethylenediamine disuccinic acid and sodium salts thereof are very widely used particularly as chelating agents.

In this specification, the abbreviation "EDDS" is used to denote the structure shown in figure 1 and said structure

in which a number of the hydroxyl hydrogen atoms have been replaced i.e., "EDDS" may also be used to refer to succinate salts in which 1, 2, 3 or 4 of the acid groups have been neutralised or partially neutralised.

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One commercially available material is trisodium ethylenediamine disuccinate. It can be purchased as an aqueous solution comprising 30 wt% EDDS (expressed as free acid) or 37 wt% of trisodium EDDS (including the counterion).

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Ethylenediamine disuccinic acid is also commercially available in the form of a solid powder. This contains 65 wt% solid [S,S] EDDS as an acid, and water of crystallisation.

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Because EDDS is an effective chelating agent, particularly of heavy metals and transition metals, it is often included in laundry and automatic dishwashing formulations.

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Trisodium EDDS is readily water soluble and thus can be incorporated into dishwashing or laundry formulations. However many consumers prefer such products in the form of powders or compressed powder tablets and as such the provision of trisodium EDDS as a liquid composition causes considerable challenges for manufacturers during formulation. Whilst solid trisodium EDDS can be isolated as a white powder, it is very hygroscopic and rapidly absorbs water from the atmosphere.

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The free tetra acid form is available in the form of a powder and so could be granulated. However it has a

solubility of only 0.3g/Kg in water which of course presents other problems with respect to its use in laundry and dishwashing applications.

- 5 There are many other situations where the lack of availability of a water soluble solid form of EDDS is a limitation to formulators of products.

The inventors have found a free flowing solid composition
10 comprising EDDS which is readily water soluble, not hygroscopic under normal atmospheric conditions and can be easily granulated.

According to a first aspect of the present invention there
15 is provided a salt of ethylenediamine disuccinic acid comprising at least 0.7 mole equivalents of magnesium per mole of ethylenediamine disuccinic acid which salt is in the form of a water-soluble, non-hygroscopic solid.

20 Preferably the salt is the form of a free flowing particulate material. It may be a powder or it may be granular.

Preferably the salt may be easily incorporated in a
25 granular composition.

In some preferred embodiments the salt comprises at least 0.8 mole of magnesium per mole of ethylenediamine disuccinic acid, preferably at least 0.9 mole, more
30 preferably at least 1 mole, preferably at least 1.3 moles, more preferably at least 1.5 moles, preferably at least 1.7 moles, more preferably at least 1.8 moles, preferably

at least 1.9 moles, and most preferably approximately 2 moles.

In some embodiments, the salt may further comprise a second alkaline earth metal, for example, calcium. It may
5 comprise up to 1.3 moles of calcium per mole of ethylenediamine disuccinic acid, for example up to 1.2 moles, for example up to 1.0 moles, up to 0.7 moles, or up to 0.5 moles.

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Especially preferred salts include dimagnesium ethylenediamine disuccinic acid, i.e. the salt having 2 moles of magnesium per mole of ethylenediamine disuccinic acid. Thus a compound in which all 4 hydroxyl hydrogen
15 atoms of figure 1 have been replaced by two magnesium ions is preferred.

Also highly preferred are salts having from 1 to 2 moles of magnesium per mole of ethylenediamine disuccinic acid.
20 For example the compound having the formula magnesium EDDS (MgEDDS) is favoured as is calcium magnesium EDDS (CaMgEDDS). The salt having 1.5 molar equivalent of magnesium per mole of ethylenediamine disuccinic acid (Mg_{1.5}EDDS) has also been found to have advantageous
25 properties.

In some embodiments the salt may include 0.05 or more moles of one or more alkali metals per mole of ethylenediamine disuccinic acid. Thus the salt may be a
30 mixed salt containing one or more alkaline earth metals and one or more alkali metals. The alkali metal when present is preferably selected from sodium, potassium and mixtures thereof. Most preferably it is sodium. In some

embodiments the salt comprises from 0.5 to 2 moles, preferably 1 to 2 moles, more preferably 1.5 to 2 moles of alkali metal per mole of ethylenediamine disuccinic acid. Thus the salt of the present invention may, for example, 5 comprise a compound of empirical formula sodium magnesium EDDS, disodium magnesium EDDS, potassium magnesium EDDS, dipotassium magnesium EDDS, or non-stoichiometric equivalents thereof. The salt of empirical formula disodium magnesium EDDS has been found to have 10 advantageous properties.

However in especially preferred embodiments, the salt of the present invention does not comprise an alkali metal. In especially preferred embodiments the salt comprises 15 only alkaline earth metal and EDDS.

The "EDDS" portion of the salt of the present invention may include any of the stereoisomers. Thus it may be selected from [R,R]-EDDS, [R,S]-EDDS, [S,S]-EDDS and any 20 combination thereof.

Preferably the salt comprises at least 50% [S,S]-EDDS, preferably at least 70%, more preferably at least 90%. In some preferred embodiments the salt consists essentially 25 of a magnesium salt of [S,S]-EDDS.

The salt of the present invention is not hygroscopic under normal atmospheric conditions. Preferably the salt of the present invention absorbs less than 10% of its weight of 30 water upon exposure to normal atmospheric conditions for 72 hours, preferably less than 5% of its weight, preferably less than 2.5%, more preferably less than 1.5% of its weight. Preferably it absorbs less than 5% of its

weight of water upon exposure to normal atmospheric conditions for 96 hours, preferably less than 3% of its weight, preferably less than 1% of its weight, more preferably less than 0.5% preferably less than 0.1% and
5 most preferably less than 0.05% of its weight. Preferably it absorbs less than 0.1% of its weight of water after exposure to normal atmospheric conditions for 14 days.

The salt of the present invention preferably has a
10 solubility which provides a concentration of EDDS of at least 8g/Kg, preferably at least 10 g/Kg in water at room temperature, preferably at least 50 g/Kg, more preferably at least 100 g/Kg, more preferably at least 125 g/Kg and most preferably at least 150 g/Kg. In some embodiments
15 the salt may have a solubility of up to 200 g/Kg, preferably up to 190 g/Kg, more preferably up to 180 g/Kg and most preferably up to 175 g/Kg.

The above solubility values refer to the weight of
20 succinate present in an aqueous solution, expressed as the weight of equivalent ethylene diamine disuccinic acid..

The solid form may include water of crystallisation. This may be present in an amount of from 5 to 45%, for example
25 from 15 to 20%.

According to a second aspect of the present invention, there is provided a composition comprising a salt of the first aspect.

30 The composition may consist essentially of the salt of the first aspect or it may include one or more further components. The composition may be a solid composition or a liquid composition. The composition may be of the form

of any composition in which trisodium EDDS or ethylenediamine disuccinic acid has previously been incorporated.

5 The composition may, for example, be a laundry composition or an automatic dishwashing composition. The composition may be in the form of a powder, for example a free flowing powder. Alternatively the composition may be in the form of compressed tablets, or encased, in liquid or solid
10 form, in a shell of a water-soluble polymeric material.

The composition may be a granular composition.

Solid laundry compositions of the present invention
15 preferably comprise from 0.01 to 10 wt%, more preferably 0.01 to 2 wt%, most preferably 0.1 to 0.5 wt% of a salt of the first aspect.

Liquid laundry compositions of the present invention
20 preferably comprise from 0.01 to 25 wt%, more preferably 0.1 to 10 wt%, most preferably 1 to 5 wt% of a salt of the first aspect.

Automatic dishwashing compositions of the present
25 invention preferably comprise 0.1 to 60 wt% of a salt of the first aspect, more preferably 1 to 30 wt% and most preferably 2 to 15 wt%.

Laundry and dishwashing compositions of the present
30 invention preferably comprise further ingredients selected from surfactants, builders, bleaches, bleach activators, redeposition additives, dye transfer inhibitors, enzymes, colorants and fragrances.

The composition of the present invention may be a bleaching composition. It may be a cleaning composition. It may be personal care composition.

According to a third aspect of the present invention there
5 is provided a method of preparing a salt according to the first aspect.

Preferably the method of the third aspect involves adding a base of magnesium to ethylenediamine disuccinic acid.
10 Preferably the method comprises adding a magnesium base to a suspension of ethylenediamine disuccinic acid, preferably an aqueous suspension. Preferably this suspension comprises from 10 to 450, for example 50 to 200 grams of acid per litre of water.

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Any suitable base can be used. For example, the base may be selected from carbonates, hydroxides, hydrides, amides and oxides. Preferably the base is magnesium hydroxide.

20 In some embodiments in which the salt comprises a mixed salt, the method of the third aspect may further comprise adding a calcium base and/or an alkali metal base to ethylenediamine disuccinic acid.

25 The present invention further provides the use of a salt of the first aspect in any manner in which EDDS-containing materials are used.

These compositions are particularly useful in paper and
30 pulp bleaching. Compositions of this aspect may also find utility in laundry and dishwashing applications. As detailed above the salt of the first aspect may also be used in other bleaching applications.

The present invention thus provides the use of the salt of the first aspect as a chelating agent. In particular the salts of the first aspect of the present invention are used as chelating agents for binding transition metals and heavy metals, for example copper, iron and manganese.

Thus the present invention includes the use of a salt of the first aspect in detergent compositions, for example laundry or automatic dishwashing compositions.

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The present invention includes the use of a salt of the first aspect in agricultural applications. For example the salt may be used in slug pellets, in herbicides, in foliar feeds, in nutrient feeds and in hydroponics.

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The present invention provides the use of a salt of the first aspect in pulp and paper bleaching. This includes mechanical bleaching and chemical bleaching as well as thermo-mechanical bleaching. The salt of the first aspect may be used in the Q stage and the P stage of the pulp bleaching, that is the wash in which metals are removed and the peroxide stage in which bleaching occurs. These terms are well understood to those skilled in the art.

25 The present invention provides the use of a salt of the first aspect in personal care applications. For example the salt may be incorporated in hair care compositions, for example hair dyes and shampoo. It may also be included as an antioxidant in creams, for example sun creams and the like.

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The present invention includes the use of a salt of the first aspect as a biocide potentiator. As such it may be

able to increase the effectiveness of a biocide and may find wide application. For example it may be used in personal care applications.

- 5 The present invention provides the use of a salt of the first aspect in household, institutional and industrial cleaning applications. It may be included in hard surface cleaners, bathroom and kitchen cleaners, in bottle washing applications or in the cleaning of dairy equipment.

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The present invention further provides the use of a salt of the first aspect as an anti-scalant material, for example as a sequestrant of calcium and magnesium salts.

- 15 The salt of the present invention may be used in oil field applications as a scale remover, for example to remove barium and strontium salts.

The present invention may provide the use of a salt of the
20 first aspect in metal cleaning applications, for example printed circuit boards or electroless plating applications.

The present invention may provide the use of a salt of the
25 first aspect in medical applications, for example, as an anti-poison material. The present invention may be used to assist the delivery of metals to parts of the body.

The present invention may provide the use of a salt of the
30 first aspect in food applications, for example as a stabiliser or antioxidant.

The invention will now be further described by way of the following non-limiting examples.

Example 1 - Synthesis of compounds

The components detailed in table 1 were prepared by the following method: In each case, 100.0g of Enviomet C320
5 (ethylene diamine disuccinic acid (65% active)) [0.2 moles] was slurried in 1L de-ionised water and the stated amount of $\text{Mg}(\text{OH})_2$ and/or $\text{Ca}(\text{OH})_2$ and/or NaOH was added. The mixture was stirred for 17 hours before being filtered. The solution was concentrated and the product
10 was allowed to crystallise out. The white crystalline product was collected by filtration and dried in vacuum oven at 40°C overnight. The EDDS content was determined by HPLC and the metal content by ICP.

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Table 1 – Preparation and Analysis of Examples

	Compound(s) added to 0.2 moles of EDDS:										
Example	Mg(OH) ₂		Ca(OH) ₂		NaOH		Analysis: (g/Kg)				Empirical Formula
	Mass (g)	Moles	Mass (g)	Moles	Mass (g)	Moles	EDDS	Mg	Ca	Na	
A	25.96	0.4					676.1	111.0			Mg _{2.0} EDDS
B	13.68	0.2					694	59.3			Mg _{1.0} EDDS
C	20.52	0.3					730	75.2			Mg _{1.2} EDDS
D (Comparative)	6.84	0.1			13.31	0.3	750	31.3		81.2	Mg _{0.5} Na _{1.4} EDDS
E	13.67	0.2	16.29	0.2			654.6	45.8	70.2		Ca _{0.8} Mg _{0.8} EDDS
F (Comparative)			32.58	0.4			723.1		199.0		Ca ₂ EDDS
G	12.98	0.2			17.81	0.4	656.7	109.7		50.8	Na _{2.1} Mg _{0.9} EDDS

Example 2

The appearance of each of the materials was observed periodically over 72 hours. The observations are recorded in table 2.

Table 2

Example	Initial Appearance	Appearance after 24hr	Appearance after 48hr	Appearance after 72hr
Na ₃ EDDS	Free flowing powder	Sticky Solid (2hr)	Very Sticky Solid	Very Sticky Solid
A	Free flowing powder	Free flowing powder	Free flowing powder	Free flowing powder
B	Free flowing powder	Free flowing powder	Free flowing powder	Free flowing powder
C	Free flowing powder	Free flowing powder	Free flowing powder	Free flowing powder
D (Comparative)	Free flowing powder	Slightly Sticky powder	Slightly Sticky powder	Slightly Sticky powder
E	Free flowing powder	Free flowing powder	Free flowing powder	Free flowing powder
F (Comparative)	Free flowing powder	Slightly Sticky powder	Slightly Sticky powder	Slightly Sticky powder
G	Free flowing powder	Free flowing powder	Free flowing powder	Free flowing powder

Table 3 show how the samples increased in mass over time.

Table 3

Example	% increase in mass after 72hr
A	0.10%
B	0.84%
C	0.46%
D (Comparative)	5.48%
E	0.32%
F (Comparative)	8.81%
G	1.26%
Na ₃ EDDS	25%

Example 3 - Solubility

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The solubility of each sample was measured as follows:

An excess of solid material was added to 5ml of water and stirred overnight at room temperature. The solution was filtered and the filtrate analysed for EDDS content by HPLC. The solubility values given in table 4 refer to the amount of free EDDS or succinate ion present and ignore the mass of any associated counter ion.

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Table 4

Example	EDDS content of a saturated solution solubility (g/kg)
EDDS.nH ₂ O	0.3
A	164.3
B	17.3
C	93.6
D (Comparative)	61.9
E	65.3
F (Comparative)	10.1
G	>300

Claims

1. A salt of ethylenediamine disuccinic acid comprising at least 0.7 mole equivalents of magnesium per mole of ethylenediamine disuccinic acid which salt is in the form of a water-soluble, non-hygroscopic solid.

2. A salt according to claim 1 which comprises at least 0.9 moles magnesium per mole of ethylenediamine disuccinic acid.

3. A salt according to claim 1 or claim 2 which comprises at least 1.8 moles of alkaline earth metal per mole of ethylenediamine disuccinic acid.

4. A salt according to any preceding claim which absorbs less than 5% of its weight of water upon exposure to standard atmospheric conditions for 72 hours.

5. A salt according to any preceding claim which has a solubility which provides a concentration of EDDS of at least 10 g/Kg.

6. A salt according to any preceding claim which is in the form of free flowing particulate material.

7. A composition comprising a salt as claimed in a preceding claim.

8. A composition as claimed in claim 7 which is a granular composition.

9. A method of preparing a salt as claimed in any of claims 1 to 6, the method comprising adding a base of an alkaline earth metal to ethylenediamine disuccinic acid.

5 10. The use of a salt as claimed in any of claims 1 to 6 improve the stability of a composition comprising hydrogen peroxide or other peroxygen-containing compound or precursor thereof.

10 11. The use of a salt as claimed in any of claims 1 to 6 as a chelating agent, an anti-scalant material or a scale remover.

12. The use of a salt as claimed in any of claims 1 to 6
15 in a detergent composition.

13. The use of a salt as claimed in any of claims 1 to 6 in personal care or cleaning applications or as a biocide potentiator.

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14. The use of a salt as claimed in any of claims 1 to 6 in agricultural, food or medical applications.

15. The use of a salt as claimed in any of claims 1 to 6
25 in pulp or paper bleaching.

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