



(86) Date de dépôt PCT/PCT Filing Date: 2002/08/30  
(87) Date publication PCT/PCT Publication Date: 2003/03/13  
(45) Date de délivrance/Issue Date: 2011/12/13  
(85) Entrée phase nationale/National Entry: 2004/03/01  
(86) N° demande PCT/PCT Application No.: EP 2002/009825  
(87) N° publication PCT/PCT Publication No.: 2003/020861  
(30) Priorité/Priority: 2001/09/04 (GB0121394.1)

(51) Cl.Int./Int.Cl. *C11D 3/33* (2006.01),  
*C11D 17/04* (2006.01)  
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(54) Titre : COMPOSITIONS POUR LE TRAITEMENT DU LINGE, DESTINEES A ETRE UTILISEES DANS UNE  
SECHEUSE A CULBUTAGE  
(54) Title: LAUNDRY COMPOSITIONS FOR USE IN A TUMBLE DRYER

(57) **Abrégé/Abstract:**

A laundry treatment process, which comprises tumble drying clothes in the presence of a chelating agent which has one nitrogen atom and at least four carboxylic acid groups per molecule, or a salt thereof. The preferred chelating agent being is a disuccinnic acid or a salt thereof of the general formula  $\text{HOOC}-(\text{R}_1.\text{COOH})\text{R}_2-\text{NR}_5-\text{R}_3(\text{R}_4.\text{COOH})-\text{COOH}$  wherein R1-R5 are preferably each independently, C1-C5 alkyl, hydroxy-alkyl or hydrogen.

## (12) INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

(19) World Intellectual Property Organization  
International Bureau



(43) International Publication Date  
13 March 2003 (13.03.2003)

PCT

(10) International Publication Number  
**WO 03/020861 A1**

(51) International Patent Classification<sup>7</sup>: **C11D 3/33**, 17/04

(21) International Application Number: PCT/EP02/09825

(22) International Filing Date: 30 August 2002 (30.08.2002)

(25) Filing Language: English

(26) Publication Language: English

(30) Priority Data:  
0121394.1 4 September 2001 (04.09.2001) GB

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(81) Designated States (*national*): AE, AG, AL, AM, AT (utility model), AT, AU, AZ, BA, BB, BG, BR, BY, BZ, CA, CH, CN, CO, CR, CU, CZ (utility model), CZ, DE (utility model), DE, DK (utility model), DK, DM, DZ, EC, EE (utility model), EE, ES, FI (utility model), FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, OM, PH, PL, PT, RO, RU, SD, SE, SG, SI, SK (utility model), SK, SL, TJ, TM, TN, TR, TT, TZ, UA, UG, US, UZ, VC, VN, YU, ZA, ZM, ZW.

(84) Designated States (*regional*): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZM, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, BG, CH, CY, CZ, DE, DK, EE, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE, SK, TR), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GQ, GW, ML, MR, NE, SN, TD, TG).

**Published:**

— with international search report

For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: LAUNDRY COMPOSITIONS FOR USE IN A TUMBLE DRYER

(57) Abstract: A laundry treatment process, which comprises tumble drying clothes in the presence of a chelating agent which has one nitrogen atom and at least four carboxylic acid groups per molecule, or a salt thereof. The preferred chelating agent being is a disuccinnic acid or a salt thereof of the general formula  $\text{HOOC}-(\text{R}_1.\text{COOH})\text{R}_2-\text{NR}_5-\text{R}_3(\text{R}_4.\text{COOH})-\text{COOH}$  wherein R1-R5 are preferably each independently, C1-C5 alkyl, hydroxy-alkyl or hydrogen.



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## LAUNDRY COMPOSITIONS FOR USE IN A TUMBLE DRYER

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Technical Field

The present invention relates to improvements to fabric treatment compositions and a process for use of the same. It is particularly concerned with a method and compositions for use with a tumble drier.

Background to the Invention

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Sequestering agents have been used in detergents since ancient times, when materials such as citric and tartaric acid were employed to obtain clear products with good solubility. In particular traces of metals such as ferric iron can cause discoloration of textiles and it has long been known to use sequestering agents, such as oxalic acid, to remove them.

In 1937, IG Farbenindustrie introduced the amino-carboxylic acid sequestrants for use in the dyeing industry. It is well known that traces of metals, particularly calcium, magnesium, iron, copper and aluminium are often responsible for faulty dyeing. These impurities can be present in a dye bath because of corrosion from fittings, from the water used or as carry over from a previous process. The difficulties that occur in the dyeing process are due to the sensitivity

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of the dyestuffs to these trivalent and divalent cations. Dyestuffs form less soluble salts with these metals and this can lead to precipitation. Additionally, many dyestuffs contain complexing groups which complex with metal ions and may lead to changes in the solubility or colour of the dye. This latter point is particularly true with the mordant dyes. Chrome dyes, on the other hand intentionally chelate chromium and the replacement of chromium with iron in the dyestuff leads to a disastrous change of colour.

10

Almost everyone is aware that repeated laundering of clothes will eventually cause most colours to fade or change in tint or hue. Consequently, many products have been marketed under the banner of 'colour care'. The first of these merely omitted bleach and other components which could damage colours. Later developments have included the incorporation of agents to prevent dye loss or dye transfer.

15

In certain ways, the environment within which laundering processes occur is similar to that in a dyeing bath. Dyed fabric is subjected to a combination of heat and water of variable purity and it is to be expected that the presence of metal ions will cause similar problems to those that occur in dyeing. In recent years, it has been suggested that sequestrants can have benefits in the laundering process.

20

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The laundering process comprises a series of unit operations of which several involve the wet treatment of fabrics. These can include pre-soak and pre-treatment processes, as well as the more commonplace washing/rinsing process. Often these washing and rinsing processes are mechanised. Optional

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processing includes machine-drying either by centrifuge or by the application of heat, as in a so-called 'tumble drier'. Treatment components can be added at various stages, for example, with detergents being added in the wash and  
5 conditioners being added in the rinse.

WO 2000/34427 discloses the use of the sequestrant IDS (iminodisuccinic acid) at various stages within the laundry process as part of a non-bleaching detergent composition  
10 containing IDS for colour care and stain-removal benefits. US 5686376 discloses the possibility of adding sequestering agents in the rinse to improve colour fidelity.

Instances of treatment in the drier are less common. Often,  
15 treatment in the drier is restricted to the addition of perfume (from a so-called 'drier sheet') although it is also possible to treat fabrics with conditioners/softeners during this operation.

20 US 6107270 discloses dryer-added fabric softener compositions that are said to provide colour and other fabric benefits. Suggested sequestering agents include TPED (Quadrol™ L), EDDS and NTA. US 6025321 discloses further dryer-added fabric softener composition to provide colour  
25 and other fabric benefits, suggesting the same sequestering agents.

Persistent staining and colour loss and/or change on fabrics are major issues for many users of domestic detergent  
30 compositions. There is therefore an ongoing need to find

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improved sequestering agents and or the means for delivering them.

5 Brief Description of the Invention

We have determined that a particular group of sequestering agents gives an improved colour benefit and indeed even a colour restoration benefit when used in the tumble drier. It  
10 is believed that this is in some way due to the removal of metal ions associated with the fabrics either through staining or through an earlier step in the laundry process.

It is believed that treatment with these materials prevents  
15 further transition metal ion colour damage and that such treatment can give stain removal benefits in the next wash.

The present invention provides a laundry treatment process, which comprises tumble drying clothes in the presence of a  
20 chelating agent which has one nitrogen atom and at least four carboxylic acid groups per molecule, or a salt thereof.

The present invention also provides a laundry drier sheet comprising a substrate and a chelating agent is of the  
25 general formula

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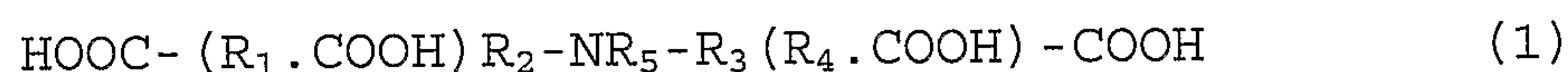
wherein  $\text{R}_1$ - $\text{R}_5$  are each independently, C1-C5 alkyl, hydroxy-  
 5 alkyl or hydrogen.

### Detailed Description of the Invention

10 Preferred 1-N 4-COOH chelating agents are disuccinnic acids containing a nitrogen hetero-atom. Preferred materials are amine-bridged.

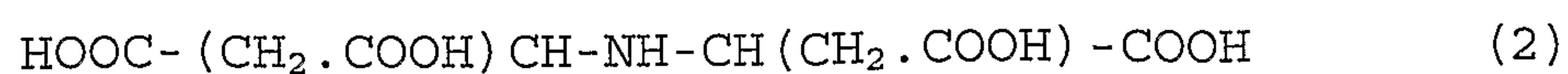
Preferably, the chelating agent is of the general formula:

15



wherein  $\text{R}_1$ - $\text{R}_5$  are each independently, C1-C5 alkyl, hydroxy-  
 alkyl or hydrogen. In this arrangement, two of the  
 20 carboxylic acid groups are disposed on either side of the bridging nitrogen atom.

In a preferred embodiment,  $\text{R}_1$ - $\text{R}_4$  in the formula (1) are methylene and  $\text{R}_5$  is hydrogen. A particularly preferred  
 25 material is therefore iminodisuccinnic acid or a salt thereof. The structure of which is given below as formula (2).



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It is envisaged that the corresponding hydroxy acids can also be used.

The process can be conducted in the presence of other fabric  
5 treatment agents. These are preferably one or more of a  
fabric softening agents, fabric conditioning agents,  
perfumes, anti-static agents, dye-transfer inhibiting  
polymers, dye fixing agents, other sequestrants, chlorine  
scavenging agents, lubricants, cross-linking polymers, soil  
10 release polymers, and optical brightening agents.

In its broadest aspect, compositions that embody the present  
invention can be in the form of a liquid, gel, paste, foam,  
powder, sheet, film, deformable solid, breakable solid or  
15 combination thereof.

A particularly preferred form is that of the drier sheet.  
Preferred sheets are non-woven, and are more preferably  
embossed. A range of suitable materials are known including  
20 a polyester/rayon (50:50 mix) in sheet form. Levels of  
sequestrant are typically such that the weight of  
sequestrant is in the range 0.0005-1.0%wt of the weight of  
cloth being placed in the drier.

25 In order that the invention may be further understood it  
will be described below with reference to the following non-  
limiting examples.

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Examples:

The reflectance versus wavelength profile of 1%, Direct Red 80 dyed, unfixed, woven, cotton fabric was measured using the large aperture on a Texicon ICS Spectraflash 500 <sup>TM</sup>, (excluding the UV below 420nm). Sixteen measurements were taken (using 4 cloth thicknesses over the Spectraflash aperture).

10 The Direct Red 80 dyed cotton fabric was obtained from Campbell Hayward Ltd. (Springvale Works, Shawclough Rd. Waterfoot, Rossendale, Lancs. BB4 9JZ) and was not made up into garments.

15 The fabric was then washed twice in demineralised water containing transition metal and water hardness ions at the following concentrations: 1.1ppm Cu<sup>2+</sup>: 2.0ppm Fe<sup>3+</sup>: 0.12ppm Mn<sup>2+</sup>: 2.3ppm Zn<sup>2+</sup>. The levels selected are quoted in the literature as being representative of the transition metal  
20 ion levels found in the main wash for soiled loads (Patent WO9403553). Water hardness was: 4°FH Ca<sup>2+</sup>: 2°FH Mg<sup>2+</sup>

Washing was performed in a vertical axis (US) machine (Whirlpool<sup>TM</sup> Super Capacity Plus Washer) set for a 12min wash  
25 on a small load setting. Two applications of transition metal ions were performed in order to damage the fabric by transition metal ion deposition. This has the effect of changing the hue of the fabric, to give a visible blue tinge. The fabric was dried in a tumble dryer (Whirlpool<sup>TM</sup>  
30 Super Capacity Dryer) after each wash.

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The change in colour of the 'colour-damaged' fabric when treated with chelating agents, provides a sensitive measure of their efficacy. For these experiments, this fabric was cut into 20cm<sup>2</sup> swatches and overlocked. The reflectance  
5 versus wavelength profile of these swatches was measured using the large aperture on a Texicon ICS Spectraflash 500 <sup>TM</sup>, (excluding the UV below 420nm) to obtain a comparative measure of damage compared to the new fabric. Here, 4 measurements were taken per swatch, using 4 cloth  
10 thicknesses over the Spectraflash aperture. Five swatches were used per drying experiment. The swatches were rewet using demineralised water, prior to mixing in with a wet ballast load, containing white non-mercerised, desized woven cotton. The ballast had previously been put through a rinse  
15 cycle with demineralised water.

Examples 1-5 and comparatives:

The dose response characteristic of IDS was investigated  
20 using the IDS concentrations (% on total weight of fabric in the load) given in table 1 below.

To obtain these results a Miele Novotronic<sup>TM</sup> T454 tumble dryer was allowed to warm up on "extra dry cotton" heat  
25 setting for 5min.

A nonwoven, embossed, polyester/rayon (50:50 mix) sheet (95gm<sup>-2</sup>) was loaded with 62.5g of IDS liquid sequestrant (Iminodisuccinic acid, tetrasodium salt, 40% activity ex.  
30 Nippon Shokubai). This corresponds to 1% IDS on weight of fabric for a 2.5kg load. The IDS solution was spread out

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evenly over the sheet, using a glass rod. The sheet was placed inside the dryer, with the 2.5kg fabric load comprising white, desized, non-mercerised, cotton ballast + five 20x20cm 'colour damaged', Direct Red 80, overlocked, 5 cotton test swatches. The load was dried on the extra dry cotton heat setting.

Once the load was dry, the test swatches were removed from the tumble dryer and the reflectance versus wavelength 10 profile was measured for each using the large aperture on a Texicon ICS Spectraflash 500 <sup>TM</sup>, (excluding the UV below 420nm). Four measurements were taken per cloth (using 4 cloth thicknesses over the Spectraflash aperture) giving a total of 20 readings per wash load. This experiment was 15 repeated for separate wash loads using concentrations of 0.5%, 0.1%, 0.01 and 0.005% IDS on weight of fabric based on a 2.5kg load. N.B. The total volume of IDS solution added onto the sheet was kept constant at 62.5g.

20 The experiment was performed twice for each IDS concentration, giving a total of 40 reflectance readings per concentration. The average reflectance reading at 620nm for each concentration is quoted in Table 1.

25 Comparison of the reflectance profiles showed that the reflectance value at 620nm was found to be the most sensitive wavelength for measuring differences in the sequestering capability of the IDS for ions on the fabric surface. The results from the dose response experiment at 30 620nm are shown below in Table 1. (95% confidence limits

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were calculated for the experiment using the Excel™ statistical analysis tool.)

5

**Table 1 - Dose Response for IDS**

| Example       | Treatment  | Mean Reflectance at 620nm | Standard Deviation | Confidence Limits |
|---------------|------------|---------------------------|--------------------|-------------------|
| Comparative A | Unwashed   | 46.56                     | -                  | -                 |
| Comparative B | Damaged    | 35.99                     | -                  | -                 |
| Comparative C | 0% IDS     | 37.92                     | 1.70               | 1.49              |
| 1             | 0.005% IDS | 39.84                     | 2.71               | 1.68              |
| 2             | 0.01% IDS  | 43.68                     | 2.72               | 1.69              |
| 3             | 0.1% IDS   | 49.64                     | 4.21               | 2.61              |
| 4             | 0.5% IDS   | 54.98                     | 0.88               | 0.54              |
| 5             | 1% IDS     | 53.89                     | 1.86               | 1.15              |

From these results it can be seen that washing fabric in the presence of the metal ions causes a significant lowering of reflectance at this wavelength (compare comparative A with B). Some of this loss can be recovered by wetting and drying the fabric with demineralised water (compare comparative B with C). However, it can be seen that the presence of even low levels of IDS causes a significant improvement in the recovery of mean reflectance.

Examples 6 and comparatives:

A level of 0.01% sequestrant was chosen for the investigation into the relative performances between the sequestrants listed below:

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- a) TPED (N, N, N', N' tetrakis (2-hydroxy propyl) ethylenediamine: available as Quadrol <sup>TM</sup> L, 100% activity, ex BASF).
- 5 b) EDDS (ethylene diamine disuccinic acid tri sodium salt: available as Octaquest E30 <sup>TM</sup> ex Octel 32.7%)
- c) NTA (Nitrilotriacetic acid sodium salt monohydrate 99%  
10 ex Aldrich).

The method used was the same as that used for examples 1-5.  
Results are given in table 2 below:

15

Table 2: - Comparison with other sequestrants

| Example          | Treatment  | Mol.<br>Wt. | Mean<br>Ref. at<br>620nm | Std.<br>Deviation | 95%<br>Conf. |
|------------------|------------|-------------|--------------------------|-------------------|--------------|
| Comparative<br>A | Unwashed   | -           | 46.56                    | 0.16              | 0.16         |
| Comparative<br>B | Damaged    | -           | 35.99                    | 0.29              | 0.28         |
| Comparative<br>C | 0% IDS     | -           | 37.92                    | 1.70              | 1.49         |
| 6                | 0.01% IDS  | 337.1       | 43.68                    | 2.72              | 1.69         |
| Comparative<br>D | 0.01% EDDS | 358         | 40                       | 3.02              | 1.87         |
| Comparative<br>E | 0.01% TPED | 292.42      | 40                       | 1.70              | 1.05         |
| Comparative<br>F | 0.01% NTA  | 257         | 39                       | 3.9               | 2.42         |

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From these results it can be shown that the IDS sequestrant shows statistically better performance at equivalent weight (0.01%) than the other sequestrants (TPED, EDDS and NTA, comparatives D-F).

5

Example 7 and Comparatives:

Woven fabric dyed with 1% Direct Red 80 (unfixed) was obtained from Campbell Hayward Ltd. (Springvale Works,  
10 Shawclough Rd. Waterfoot, Rossendale, Lancs. BB4 9JZ)

The reflectance versus wavelength profile was measured for the new fabric using the large aperture on a Texicon ICS Spectraflash 500 <sup>TM</sup>, (excluding the UV below 420nm). Sixteen  
15 measurements were taken (using 4 cloth thicknesses over the Spectraflash aperture).

The red woven fabric was treated with transition metal ions as follows:

20

The fabric was washed twice in demineralised water containing transition metal and water hardness ions at the following concentrations: 1.1ppm Cu<sup>2+</sup>: 2.0ppm Fe<sup>3+</sup>: 0.12ppm Mn<sup>2+</sup>: 2.3ppm Zn<sup>2+</sup>. The levels selected are quoted in  
25 literature (WO9403553) as being representative of the transition metal ion levels found in the main wash when soiled loads are used. Water hardness was: 4°FH Ca<sup>2+</sup>: 2°FH Mg<sup>2+</sup>

30 Washing was performed in a vertical axis (US) machine (Whirlpool<sup>TM</sup> Super Capacity Plus Washer) set for a 12min wash

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on a small load setting. Two applications of transition metal ions were performed in order to damage the fabric by transition metal ion deposition. This has the effect of changing the hue of the fabric, to give a visible blue  
5 tinge. The fabric was dried in a tumble dryer (Whirlpool™ Super Capacity Dryer) in between each wash.

The change in colour of the 'colour-damaged' fabric when treated with chelating agents, provides a sensitive measure  
10 of their efficacy. For these experiments, this fabric was cut into 20cm<sup>2</sup> swatches and overlocked. The reflectance of these swatches was measured to obtain a comparative measure of damage. The reflectance versus wavelength profile was measured for the 'colour-damaged' fabric using the large  
15 aperture on a Texicon ICS Spectraflash 500™, (excluding the UV below 420nm). Sixteen measurements were taken (using 4 cloth thicknesses over the Spectraflash aperture)

Six (20x20cm) overlocked swatches of the 'colour-damaged'  
20 woven fabric dyed with 1% Direct Red 80 (unfixed) were taken, dampened with demineralised water and mixed randomly into a 2.5kg ballast load (white, desized, non-mercerised woven cotton sheeting) which had previously been put through a rinse cycle with demineralised water.

25

Sequestrants used were:

TPED: N, N, N', N' tetrakis (2-hydroxy propyl)  
ethylenediamine: available as Quadrol™ L, 100%  
30 activity, ex BASF.

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EDDS: Ethylene diamine disuccinic acid tri sodium salt:  
available as Octaquest E30 <sup>TM</sup> ex Octel 32.7%.

NTA: Nitritotriacetic acid sodium salt monohydrate 99% ex  
Aldrich.

IDS: Iminodisuccinic acid tetra sodium salt (ex Nippon  
Shokubai Co. Ltd 40.2% aqueous solution)

10 Moles of all sequestrants used in experiment =  $2.14 \times 10^{-3}$  moles.

A 50ml solution of  $2.14 \times 10^{-3}$  moles IDS in demineralised water  
was prepared. A nonwoven, embossed, polyester/rayon (50:50  
mix) sheet ( $95 \text{ gm}^{-2}$ ) was loaded with the 50g IDS sequestrant  
15 solution described above. The IDS solution was spread out  
evenly over the sheet, using a glass rod.

A US Whirlpool<sup>TM</sup> Super Capacity Dryer was allowed to warm up  
for 5 minutes on permanent press/heavy setting. The tumble  
20 dryer sheet that had been loaded with IDS was then placed  
inside the dryer, with the 2.5kg fabric load (ballast + 6  
test swatches). The load was dried using the permanent  
press/heavy setting for 90 minutes.

25 The process was repeated again with a separate, identical  
load.

The entire regime was repeated with the other sequestrants,  
TPED, NTA and EDDS.

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The reflectance versus wavelength profile was measured for the treated fabrics using the large aperture on a Texicon ICS Spectraflash 500 <sup>TM</sup>, (excluding the UV below 420nm). Four measurements were taken for each piece of fabric in each load using 4 cloth thicknesses over the Spectraflash aperture. This gave 24 measurements per load and 48 readings per treatment.

The reflectance value of the coloured fabric at 620nm is the most sensitive to the effects of transition metal ions in this system. The changes in reflectance at 620nm for the fabric after treatment with the various sequestrants are shown below (in table 3) for comparison.

**Table 3: Comparison with other sequestrants at equimolar concentration ( $2.14 \times 10^{-3}$  moles).**

| Treatment | Mean reflectance<br>at 620nm | Standard<br>Deviation | 95% confidence<br>Limits |
|-----------|------------------------------|-----------------------|--------------------------|
| Unwashed  | 46.56                        | 0.16                  | 0.16                     |
| Damaged   | 35.99                        | 0.29                  | 0.28                     |
| IDS       | 44.03                        | 4.48                  | 1.27                     |
| TPED      | 38.23                        | 1.11                  | 0.31                     |
| EDDS      | 39.41                        | 2.99                  | 0.85                     |
| NTA       | 38.45                        | 1.71                  | 0.48                     |

From these figures it can be seen that the IDS containing system is significantly better than the other materials at the concentration used.

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**CLAIMS**

1. Laundry treatment process, which comprises tumble drying clothes in the presence of a chelating agent which has one nitrogen atom and at least four carboxylic acid groups per molecule, or a salt thereof.
2. Process according to claim 1, wherein the chelating agent is a disuccinnic acid or a salt thereof.
3. Process according to claim 2, wherein the disuccinnic acid is amine bridged.
4. Process according to claim 1, wherein the chelating agent is of the general formula



wherein  $\text{R}_1$ - $\text{R}_4$  are each independently  $\text{C}_1$ - $\text{C}_4$  alkylene or hydroxy-alkylene, and wherein  $\text{R}_5$  is  $\text{C}_1$ - $\text{C}_5$  alkyl, hydroxy-alkyl or hydrogen.

5. Process according to claim 4, wherein  $\text{R}_1$ - $\text{R}_4$  are methylene and  $\text{R}_5$  is hydrogen.
6. Process according to any one of claims 1 to 5, conducted in the presence of one or more of a fabric softening agent, fabric conditioning agent, perfume, anti-static agent, dye-transfer inhibiting polymer, dye fixing agent, other sequestrant, chlorine

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scavenging agent, lubricants, cross-linking polymers and optical brightening agents.

7. A laundry drier sheet comprising a substrate and a chelating agent of the general formula



wherein  $\text{R}_1-\text{R}_4$  are each independently  $\text{C}_1-\text{C}_4$  alkylene or hydroxy-alkylene, and wherein  $\text{R}_5$  is  $\text{C}_1-\text{C}_5$  alkyl, hydroxy-alkyl or hydrogen.