

(19)



LE GOUVERNEMENT
DU GRAND-DUCHÉ DE LUXEMBOURG
Ministère de l'Économie

(11)

N° de publication :

LU502047

(12)

BREVET D'INVENTION**B1**

(21)

N° de dépôt: LU502047

(51)

Int. Cl.:

G01N 31/22, G01N 21/78

(22)

Date de dépôt: 07/05/2022

(30)

Priorité:

(72)

Inventeur(s):

VAN GURP Lennard - Pays-Bas

(43)

Date de mise à disposition du public: 07/11/2023

(74)

Mandataire(s):

Biopatents IP Consultancy - 5691NW Son (Pays-Bas)

(47)

Date de délivrance: 07/11/2023

(73)

Titulaire(s):

SEEF B.V. - 4851 SJ Ulvenhout (Pays-Bas)

(54)

Method for the detection of chromium-6 in a sample.

(57)

Disclosed is a method for determination of the presence chromium-6 (Cr-6) in a solid sample, comprising the steps of contacting the solid sample with a destruction liquid under conditions wherein Cr-6 from the solid sample dissolves in the destruction liquid as a chromate, providing a test solution; subjecting the test solution obtained in step (1) to a specific Cr-6 detection reaction, under conditions wherein the presence of Cr-6 is detected; and detecting whether the Cr-6 specific reaction occurred in step (2); wherein the destruction liquid comprises an oxidator, having a higher standard electrode potential E^0 than the chromate.

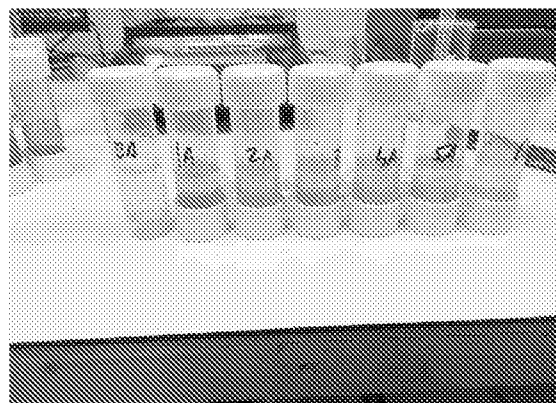


Fig. 1

The invention relates to a method for determination of the presence chromium-6 (Cr-6) in a solid sample, comprising the steps of contacting the solid sample with a destruction liquid under conditions wherein Cr-6 from the solid sample dissolves in the destruction liquid as a chromate, providing a test solution; subjecting the said test solution to a specific Cr-6 detection reaction, under conditions wherein the presence of Cr-6 is detected; and detecting whether the Cr-6 specific reaction occurred.

For over 70 years, hexavalent chromium, herein also chromium-6, has been widely used as anti-corrosive agent in paints and primers, especially as a spray paint on metal surfaces, ships and aircraft, defence equipment, infrastructure objects, factories, containers, and many types of metal structures, such as steel window frames. However, chromium-6 is toxic, and the use thereof has now been abandoned.

However, many painted surfaces may or may not contain chromium-6. In order to check whether a painted surface contains chromium or not, several tests have developed, in particular based on method 7600 of the NIOSH Manual of Analytical Methods (NMAM) of August 15, 1994.

According to this method, the solid sample is obtained, e.g., by scratching off some pieces of paint from a painted metal surface and treating these in a destruction liquid to bring the hexavalent chromium in solution as a chromate. This can be done in a strong acid destruction medium, wherein the chromate is predominantly present as the dichromate ion $\text{Cr}_2\text{O}_7^{2-}$, or in a strong basic destruction medium, wherein the chromate is predominantly present as the monochromate ion CrO_4^{2-} .

Subsequently, the chromate solution is subjected to a specific Cr-6 detection reaction wherein a colouring reaction with a chromogenic agent, in particular 1,5-diphenylcarbazide is performed in acid conditions.

The problem of the methods, known in the art is that during destruction of the sample, the hexavalent Cr-6 will oxidise any metallic contaminations in the sample, such as aluminium or zinc, resulting in the formation of trivalent chromium, Cr-3, and the aluminium or zinc oxide, or both.

The trivalent chromium is not capable of reacting with the chromogenic agent and as a result, the test results are unreliable since not all Cr-6 will be detected, or even not detected at all, although it was present in the paint sample.

Since the dichromate is an even stronger oxidator than the monochromate, the destruction liquid for such solid paint samples is usually a basic destruction liquid, but still, the risk for false negative test results (i.e. wherein falsely no Cr-6 or a lower content thereof is detected) is significant.

The present invention now provides a very elegant solution to the above problem and is thereto characterized in that the destruction liquid comprises an oxidator, having a higher standard electrode potential E^0 than the chromate. The chromate here is the chromate that is dissolved in the destruction liquid. Since both mono – and dichromates are in equilibrium with one another, both chromates will usually be present in the destruction liquid. Under highly acidic and concentrated conditions, higher chromates such as trichromate ($\text{Cr}_3\text{O}_{10}^{2-}$), tetrachromate ($\text{Cr}_4\text{O}_{13}^{2-}$) and polychromates may also be formed.

A stronger oxidator will immediately react with any available electron, since it is a stronger oxidator than the chromate. Even if the chromate would tend to be reduced to Cr-3, i.e., in a soluble hydroxide, the strong oxidator will immediately oxidise the said Cr-3 back to Cr-6, so that any possibly false negatives (the Cr-3 originating from Cr-6) are returned into the proper and accurate true positive.

Any Cr-3 that may have been originally present in the sample will however not be oxidised to Cr-6, since the Cr-3 in a paint sample would be present as Cr_2O_3 . The reaction surface of Cr_2O_3 is limited and Cr_2O_3 does not dissolve sufficiently well to be vulnerable to oxidation to CrO_4^{2-} , even in the presence of a strong oxidator as defined herein (an oxidator, having a higher standard electrode potential E^0 than the chromate), so that false positive results are neglectable.

For a proper destruction of the solid Cr-6, the destruction liquid is preferably chosen from an acid destruction liquid having a pH of 1 – 2, and a basic destruction liquid having a pH of 12 – 14. Since the destruction liquid comprises a strong oxidator, stronger than the chromate originating from the sample, it is now very well possible to use an acid destruction liquid.

To this end, the acid destruction liquid preferably comprises H_2SO_4 , HNO_3 or HCl , whereas the alkaline destruction liquid preferably comprises NaOH or KOH and Na_2CO_3 .

Nevertheless, the use of a basic destruction liquid is slightly preferred since the E^0 of the monochromate predominant at basic pH, is slightly lower than the E^0 of the dichromate, predominant at acidic pH.

Therefore, in an attractive embodiment, the destruction liquid is a basic destruction liquid comprising 0.05 – 1 M NaOH , preferably 0.4 – 0.6 M NaOH , and preferably further comprises 0.03 – 0.6 M Na_2CO_3 , preferably 0.2 – 0.4 M Na_2CO_3 .

The oxidator is preferably chosen such, that the standard electrode potential E^0 thereof is above 1.23 mV, the E^0 of the dichromate. The oxidator in the destruction liquid is preferably chosen from the group, consisting of hydrogen peroxide (E^0 of 1.78), manganese dioxide (E^0 of 1.24), persulfate (E^0 of 2.01), hypochlorite (E^0 of 1.63), permanganate (E^0 of 1.49), chlorine (E^0 of 1.36), cerium-4 (E^0 of 1.44), ozone (E^0 of 2.08) and fluorine (E^0 of 2.87).

The destruction liquid preferably comprises 1 mM – 1 M of the oxidator, more preferably 7 – 160 mM, even more preferably 10 – 100 mM. It was found that with such an amount of oxidator, the Cr-6 present in the sample can reliably be detected both qualitatively and quantitatively. LU502047

Although the skilled person is aware of alternative detection reaction capable of specifically detecting the presence of dissolved Cr-6 (i.e., in the form of a chromate), the specific Cr-6 detection reaction preferably comprises a chromogenic agent capable of specifically reacting with a chromate resulting in a colour change, the chromogenic agent preferably being chosen from 1,5-diphenylcarbazide and variamine blue B. The specific detection reaction of the NIOSH7600 standard involves a colouring reaction with 1,5-diphenylcarbazide.

In another aspect, the invention relates to a destruction liquid as described above. the destruction liquid preferably comprises

- 0.4 – 0.6 M NaOH
- 0.2 – 0.4 M Na₂CO₃.
- 10 – 100 mM of an oxidator having a standard electrode potential E^0 of more than 1.23 mV.

The said oxidator is preferably any of the oxidants as described or preferred above, and in particular comprises hydrogen peroxide.

In still another aspect, the invention relates to a kit, comprising a first container comprising the destruction liquid as described above, and a second container comprising a reaction mixture for performing the detection reaction as described above. The kit preferably further comprises a third container comprising a positive control sample comprising a chromate, in an amount that is detectable by performing the detection reaction using the reaction mixture from the second container. To this end, the kit preferably comprises instructions for use, wherein the reaction conditions for the destruction and the detection reaction are given.

The invention will now be further explained by the following examples.

Examples

Materials:

H ₂ O ₂	: 27 w/w% aqueous solution stabilised solution, Thermo Scientific, US
KMnO ₄	: potassium permanganate 99.0-100.5%, ACS, for analysis, Merck, US
NaOH	: sodium hydroxide pellets pure, Merck
Na ₂ CO ₃	: sodium carbonate ≥98%, purified, VWR Chemicals, US
H ₂ SO ₄	: sulfuric acid ≥95%, technical, VWR Chemicals
Zinc	: zinc 97,5%, powder 4 - 7 µm, Thermo Scientific
Aluminium	: aluminium, powder for synthesis, Merck
Cr-6 pigment	: potassium chromate K ₂ CrO ₄ . 99.5% for analysis, Thermo Scientific
Cr-3 pigment	: chromium(III) oxide, Technipur®, Merck

Detection reagent : 1,5-Diphenylcarbazide $\geq 97.0\%$ analytical reagent, VWR Chemicals LU502047
 Cr-6 Paint low : Roboton EP Roller Coat SF, 20 mg/kg lead chromate, Baril Coatings, Netherlands
 Cr-6 Paint high : Roboton EP Roller Coat SF, 1000 mg/kg lead chromate, Baril Coatings
 5 Aluminium paint : Roboton EP Roller Coat SF, Baril Coatings
 Zinc Paint : SteelKote EP ZN HS, 50-75% Zinc content, Baril Coatings

Example 1 Titration of the oxidator

10 The composition of Samples 1-1A – 1-6A and control 1-BA are given in table 1. The ingredients are mixed together, and water was added to a final volume of 10 ml. At first sodium hydroxide was dissolved in 5 ml deionized water. After dissolution, different hydrogen peroxide volumes were added at 1-1A to 1-6A, followed by the addition of a potassium chromate solution and zinc powder. At the end deionized water was added to make a total volume of 10 ml.

15 Table 1

Sample	H ₂ O ₂ (27 w/v%)		K ₂ CrO ₄ 1000 µg/ml	NaOH		Zinc
	µl	mM		g	M	
1-BA	0	0	0.1	0.2	0.5	1.0
1-1A	10	8	0.1	0.2	0.5	1.0
1-2A	25	20	0.1	0.2	0.5	1.0
1-3A	50	40	0.1	0.2	0.5	1.0
1-4A	100	80	0.1	0.2	0.5	1.0
1-5A	200	160	0.1	0.2	0.5	1.0
1-6A	1000	800	0.1	0.2	0.5	1.0

The samples were incubated for 1 hour at 90°C, during incubation, the samples were vortexed 8 times at regular intervals for 10 seconds.

20 The sample was filtered with a (0.5 µm PTFE filter) and to each sample, 0.5 ml of the detection agent was added. The total volume was brought to 25 ml with 0.5N H₂SO₄. The colouring reaction was performed for at least 30 minutes at 20°C.

The results of the colouring reaction are shown in figure 1. It is observed that a well visible colour reaction occurs in the presence of the oxidator, whereas in absence, no colouration occurs. This means that in absence of the oxidator, all Cr-6 present has been reduced to Cr-3
 25 due to the oxidation of the zinc present. High oxidator concentrations do not further contribute to the colouring reaction. The preferred range is 7 – 160 mM, preferably 10 – 100 mM.

Example 2A Titration NaOH

30 Samples were prepared and treated as described in example 1. The composition of Samples 2-1B – 2-6B are given in table 2A.

The results of the colouring reaction are shown in figure 2. It is observed that a well visible colour reaction occurs at a pH of above 12, in particular 12.7, corresponding with a NaOH concentration of 0.5 M. Below this value, the colouration reaction is less intense. The preferred pH range is therefore 12 - 14.

LU502047

5

Table 2A

Sample	H ₂ O ₂ (27 w/v%)		K ₂ CrO ₄ 1000 µg/ml	NaOH		Zinc	pH
	µl	mM	ml	mg	M	g	
2-1B	10	80	0.1	20	0.05	1.0	12.7
2-2B	25	40	0.1	200	0.5	1.0	13.7
2-3B	50	40	0.1	200	0.5	1.0	13.7
2-4B	100	80	0.1	400	1.0	1.0	14
2-5B	200	160	0.1	600	1.5	1.0	14.2
2-6B	1000	800	0.1	800	2.0	1.0	14.3

Example 2B Titration H₂SO₄

The composition of Samples 2-1S – 2-6S are given in table 2B. The envisaged amount of sulfuric acid was added to a 5 ml solution of deionized water, followed by the addition of potassium chromate, followed by the addition of potassium permanganate and finally the zinc powder. The samples were incubated for 1 hour at 90°C. After filtration the reagent solution was added as described for example 1 to make the Cr-6 visible.

15

Table 2B

Sample	KMnO ₄ (99,5%)		K ₂ CrO ₄ (99,9%) 1000 µg/ml	H ₂ SO ₄ (99,5%)		Zinc	pH
	µg/ml	mM	ml	mg	mM	g	
2-1S	320	2	0.1	490	500	1.0	0.3
2-2S	320	2	0.1	49	50	1.0	1.3
2-3S	320	2	0.1	4,9	5	1.0	2.3
2-4S	320	2	0.1	0.49	0.5	1.0	3.3
2-5S	320	2	0.1	0.245	0.25	1.0	3.6
2-6S	320	2	0.1	0.049	0.05	1.0	4.3

It was observed that a well visible colour reaction occurs in at a pH of below 2.3, corresponding with a H₂SO₄ concentration of 5 mM. Above this value, the colouration reaction is less intense. The preferred pH range is therefore 1.0 – 2.5, more preferably 1.0 – 2.0.

20

Example 3 Reaction times

LU502047

Samples were prepared and treated as described in example 1, except for the incubation time, which is indicated in table 3, together with the composition of Samples 3-1C – 3-8C

5

Table 3

Sample	H ₂ O ₂ (27 w/v%)		K ₂ CrO ₄ 1000 µg/ml	NaOH		Zinc	Time
	µl	mM		g	M		
3-1C	0	0	0.1	0.2	0.5	1.0	5
3-2C	10	8	0.1	0.2	0.5	1.0	5
3-3C	0	0	0.1	0.2	0.5	1.0	15
3-4C	10	8	0.1	0.2	0.5	1.0	15
3-5C	0	0	0.1	0.2	0.5	1.0	30
3-6C	10	8	0.1	0.2	0.5	1.0	30
3-7C	0	0	0.1	0.2	0.5	1.0	60
3-8C	10	8	0.1	0.2	0.5	1.0	60

The results of the colouring reaction are shown in figure 3. It is observed that a visible reaction is already observed after 5 minutes incubation at 90°C.

10 Example 4 Paint samples

Known Cr-6 paint samples also have been investigated. This was done by investigating the effect of different paint layers (Cr-6 primer, zinc intermediate coating, aluminium topcoat), with and without the addition of an oxidant. Two primers were investigated, which differ in the Cr-6 content, namely plate B2, coated with a primer containing 20 mg/kg lead chromate, and plate B6, coated with a coating containing 1000 mg/kg lead chromate. Apart from the concentration of lead chromate, plates B2 and B6 are identical. The Cr-6 determination has been performed at the following conditions, wherein reference is made to example 1: extraction for 30 minutes in 10 ml NaOH/Na₂CO₃ at 90°C, 8 times vortexed for 10 seconds at regular intervals, and quantification by UV-VIS spectrometry at wavelength 543 nm (model Jenway 6300, Cole-Parmer, UK).

Table 4

Sample	H ₂ O ₂ (27 w/v%)		Cr-6 mg/kg	NaOH		Plate type	Paint layers
	µl	mM		g	M		
4-1D	0	0	6	0.2	0.5	B2	Cr-6
4-2D	15	12	6	0.2	0.5	B2	Cr-6
4-3D	0	0	0	0.2	0.5	B2	Cr-6 / Zn
4-4D	15	12	2	0.2	0.5	B2	Cr-6 / Zn
4-5D	0	0	0	0.2	0.5	B2	Cr-6 / Zn / Al
4-6D	15	12	3	0.2	0.5	B2	Cr-6 / Zn / Al

4-7D	0	0	107	0.2	0.5	B2	Cr-6
4-8D	15	12	101	0.2	0.5	B6	Cr-6
4-9D	0	0	0	0.2	0.5	B6	Cr-6 / Zn
4-10D	15	12	5	0.2	0.5	B6	Cr-6 / Zn
4-11D	0	0	0	0.2	0.5	B6	Cr-6 / Zn / Al
4-12D	15	12	8	0.2	0.5	B6	Cr-6 / Zn / Al

LU502047

Example 5 Field test

Paint samples were taken from painted bridge parts (Oranjebrug, Schiedam, The Netherlands) that were painted with different paints.

5 Samples were taken, with an amount of one gram of paint per sample, which was further analysed by XRF analysis. The paint was crushed into smaller particles followed by a XRF analysis. The XRF analysis was done by a Niton XL5 Plus. In order to get a total screening of metals the analysis was performed by light, low and main wave lengths with 30 seconds for each wavelength.

10 Table 5 shows the content of Cr-6, zinc and aluminium in the samples.

Table 5

Sample	H ₂ O ₂ (27 w/v%)		NaOH		Cr-6	Zn	Al
	μl	mM	g	M	mg/kg	mg/kg	mg/kg
5-005-	0	0	0.2	0.5	846	41799	2591
5-005+	100	80	0.2	0.5			
5-006-	0	0	0.2	0.5	1381	21241	3065
5-006+	100	80	0.2	0.5			
5-007-	0	0	0.2	0.5	1048	25734	4227
5-007+	100	80	0.2	0.5			

15 Sample 5-005 was taken from the upper bridge surface, sample 5-006 was taken from the bridge barrier, and sample 5-007 from the control panel.

20 Samples of the paint samples were prepared by weighing 1000 mg of the paint sample in a 10 ml reaction tube, and the ingredients were added as indicated in table 5. The results are shown in figure 4. It is clear that the samples containing the oxidant (samples 005, 006 and 007 on the right, in the table indicated as '+' samples) give a strong colouration reaction, whereas the samples without oxidant (samples 005, 006 and 007 on the left, in the table indicated as '-' samples) do not show any discolouration, meaning that in the samples without oxidant, the Cr-6 present has been reduced to Cr-3, whereas the samples including the oxidant show the presence of Cr-6.

CLAIMS

1. Method for determination of the presence chromium-6 (Cr-6) in a solid sample, comprising
5 the steps of
 - (1) contacting the solid sample with a destruction liquid under conditions wherein Cr-6 from the solid sample dissolves in the destruction liquid as a chromate, providing a test solution;
 - (2) subjecting the test solution obtained in step (1) to a specific Cr-6 detection reaction,
10 under conditions wherein the presence of Cr-6 is detected;
 - (3) detecting whether the Cr-6 specific reaction occurred in step (2);characterized in that the destruction liquid comprises an oxidator, having a higher standard electrode potential E^0 than the chromate.
- 15 2. Method of claim 1, wherein the destruction liquid is chosen from an acid destruction liquid having a pH of 1 – 2, and a basic destruction liquid having a pH of 12 – 14.
3. Method of claim 1 or 2, wherein
 - the acid destruction liquid comprises H_2SO_4 , HNO_3 or HCl ;
 - 20 – the acid destruction liquid comprises $NaOH$ or KOH and Na_2CO_3
4. Method of any of the claims 3 - 6, wherein the destruction liquid is a basic destruction liquid comprising 0.05 – 1 M $NaOH$, preferably 0.4 – 0.6 M $NaOH$.
- 25 5. Method of claim 7, wherein the destruction liquid comprises 0.03 – 0.6 M Na_2CO_3 , preferably 0.2 – 0.4 M Na_2CO_3 .
6. Method of any of claims 1- 3, wherein the oxidator in the destruction liquid is chosen such, that the standard electrode potential E^0 thereof is above 1.23 mV.
- 30 7. Method of claim 4, wherein the oxidator in the destruction liquid is chosen from the group, consisting of hydrogen peroxide, manganese dioxide, persulfate, hypochlorite, permanganate, chlorine, lead oxide, cerium-4, ozone and fluorine, the oxidator preferably comprising hydrogen peroxide.
- 35 8. Method of any of the preceding claims, wherein the destruction liquid comprises 1 mM – 1 M of the oxidator, preferably 10 – 100 mM.

9. Method of any of the preceding claims, wherein the specific Cr-6 detection reaction comprises a chromogenic agent capable of specifically reacting with a chromate resulting in a colour change.

5

10. Method of claim 5, wherein the chromogenic agent is chosen from 1,5-diphenylcarbazide and variamine blue B.

11. Destruction liquid as defined in any of the preceding claims.

10

12. Destruction liquid of claim 11, comprising

- 0.4 – 0.6 M NaOH
- 0.2 – 0.4 M Na₂CO₃.
- 10 – 100 mM of an oxidator having a standard electrode potential E^0 of more than 1.23 mV.

15

13. Destruction liquid of claim 11 or 12, wherein the oxidator comprises hydrogen peroxide.

14. Kit, comprising a first container comprising the destruction liquid of any of claims 11 - 13, and a second container comprising a reaction mixture for performing the detection reaction of step (2) of claim 1.

20

15. Kit of claim 14, further comprising a third container comprising a positive control sample comprising a chromate.

1. Verfahren zur Bestimmung des Vorhandenseins von Chrom-6 (Cr-6) in einer festen Probe, umfassend die Schritte
 - (1) Inkontaktbringen der festen Probe mit einer Zerstörungsflüssigkeit unter Bedingungen, bei denen sich Cr-6 aus der festen Probe in der Zerstörungsflüssigkeit als Chromat auflöst, wobei eine Testlösung bereitgestellt wird;
 - (2) Unterziehen der in Schritt (1) erhaltenen Testlösung einer spezifischen Cr-6-Nachweisreaktion unter Bedingungen, bei denen die Anwesenheit von Cr-6 nachgewiesen wird;
 - (3) Nachweis, ob die spezifische Cr-6-Reaktion in Schritt (2) stattgefunden hat; dadurch gekennzeichnet, dass die Zerstörungsflüssigkeit einen Oxidator umfasst, der ein höheres Standardelektrodenpotential E^0 als das Chromat aufweist.
2. Verfahren nach Anspruch 1, wobei die Zerstörungsflüssigkeit ausgewählt ist aus einer sauren Zerstörungsflüssigkeit mit einem pH-Wert von 1 - 2 und einer basischen Zerstörungsflüssigkeit mit einem pH-Wert von 12 - 14.
3. Verfahren nach Anspruch 1 oder 2, wobei
 - die saure Zerstörungsflüssigkeit H_2SO_4 , HNO_3 oder HCl enthält;
 - die basische Zerstörungsflüssigkeit $NaOH$ oder KOH , und Na_2CO_3 enthält
4. Verfahren nach einem der Ansprüche 1 bis 3, wobei die Zerstörungsflüssigkeit eine basische Zerstörungsflüssigkeit ist, die 0,05 - 1 M $NaOH$, vorzugsweise 0,4 - 0,6 M $NaOH$, enthält.
5. Verfahren nach Anspruch 3 oder 4, wobei die Zerstörungsflüssigkeit 0,03 - 0,6 M Na_2CO_3 , vorzugsweise 0,2 - 0,4 M Na_2CO_3 , umfasst.
6. Verfahren nach einem der Ansprüche 1 bis 5, wobei das Oxidationsmittel in der Zerstörungsflüssigkeit so gewählt wird, dass das Standardelektrodenpotential E^0 über 1,23 mV liegt.
7. Verfahren nach Anspruch 6, wobei der Oxidator in der Zerstörungsflüssigkeit aus der Gruppe ausgewählt wird, die aus Wasserstoffperoxid, Mangandioxid, Persulfat, Hypochlorit, Permanganat, Chlor, Bleioxid, Cer-4, Ozon und Fluor besteht, wobei der Oxidator vorzugsweise Wasserstoffperoxid umfasst.

8. Verfahren nach einem der vorhergehenden Ansprüche, wobei die Zerstörungsflüssigkeit 1 mM - 1 M des Oxidationsmittels, vorzugsweise 10 - 100 mM, enthält. LU502047

9. Verfahren nach einem der vorhergehenden Ansprüche, wobei die spezifische Cr-6-Nachweisreaktion ein chromogenes Mittel umfasst, das in der Lage ist, spezifisch mit einem Chromat zu reagieren, was zu einem Farbwechsel führt.

10. Verfahren nach Anspruch 9, wobei das chromogene Mittel ausgewählt wird aus 1,5-Diphenylcarbazon und Variaminblau B.

11. Zerstörungsflüssigkeit nach einem der vorhergehenden Ansprüche.

12. Zerstörungsflüssigkeit nach Anspruch 11, umfassend

- 0,4 - 0,6 M NaOH
- 0,2 - 0,4 M Na₂CO₃.
- 10 - 100 mM eines Oxidators mit einem Standardelektrodenpotential E₀ von mehr als 1,23 mV.

13. Zerstörungsflüssigkeit nach Anspruch 11 oder 12, wobei das Oxidationsmittel Wasserstoffperoxid umfasst.

14. Kit, umfassend einen ersten Behälter, der die Zerstörungsflüssigkeit nach einem der Ansprüche 11 bis 13 enthält, und einen zweiten Behälter, der ein Reaktionsgemisch zur Durchführung der Nachweisreaktion nach Schritt (2) von Anspruch 1 enthält.

15. Kit nach Anspruch 14, ferner umfassend einen dritten Behälter, der eine positive Kontrollprobe enthält, die ein Chromat umfasst.



Fig. 1

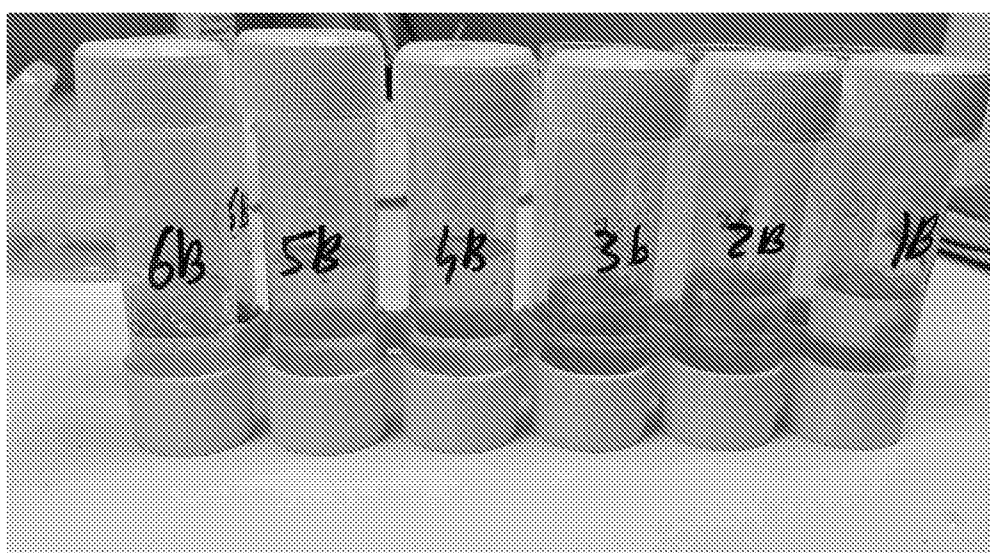


Fig. 2

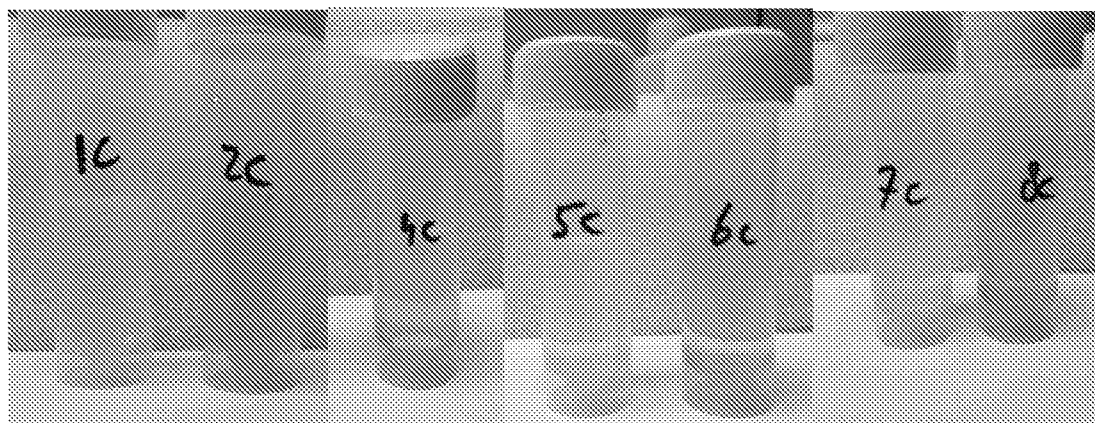


Fig. 3

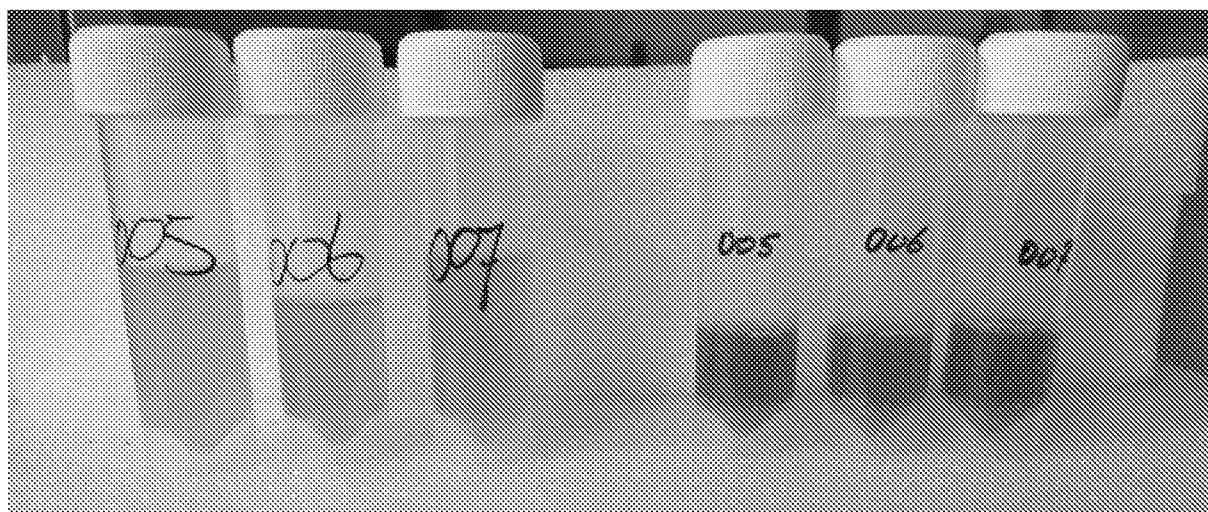


Fig. 4