
International Standard



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Paints and varnishes — Preparation of acid extracts from paints in liquid or powder form

Peintures et vernis — Préparation des extraits acides des peintures liquides ou en poudre

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Foreword

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Contents	Page
0 Introduction	1
1 Scope and field of application	1
2 References	1
3 Definitions	1
3.1 Pigment	1
3.2 "Soluble" metal content of liquid paint	1
3.3 "Soluble" metal content of paint in powder form	1
4 Principle	1
5 Sampling	2
6 Separation of the pigment from the liquid paint sample	2
6.1 Preparation of the test sample	2
6.2 Reagents	2
6.3 Apparatus	2
6.4 Procedure	2
6.4.1 Method A (for solvent-thinned paints, printing inks and similar products)	2
6.4.2 Method B (for emulsion paints based on aqueous polymer dispersions)	3
6.4.3 Method C [for plastisols and organosols based on polyvinyl chloride (PVC) and its copolymers and for paints based on other non-aqueous polymer dispersions]	3
6.5 Blank test solution	3
7 Treatment of paints in powder form	3
8 Acid extraction of the separated pigment and of paints in powder form	3
8.1 Treatment of the separated pigment	3
8.2 Method for the acid extraction of "soluble" metals [including lead when the total lead content of the paint is less than 1 % (<i>m/m</i>) (mass : volume ratio 1 : 15)	3

8.3	Method for the acid extraction of "soluble" lead [when the total lead content of the paint equals or exceeds 1 % (<i>m/m</i>)] (mass : volume ratio 1 : 1 000)	4
8.3.1	Preliminary determination of total lead	4
8.3.2	Extraction procedure	5
8.4	Blank test solution	5
9	Treatment of the extracted liquid portion	5

Paints and varnishes — Preparation of acid extracts from paints in liquid or powder form

0 Introduction

This International Standard is for use in conjunction with the appropriate parts of ISO 3856.

1 Scope and field of application

This International Standard describes methods for the preparation of acid extracts required as the test solutions for the determination of the "soluble" metal contents of paints and related products in liquid or powder form.

The acid extracts are prepared in 0,07 mol/l hydrochloric acid, which is chosen as an approximation of the acid in the stomach.

This International Standard is not applicable to dried or comminuted paint films (see ISO 6714).

WARNING — The procedures described in this International Standard are intricate and, therefore, should be carried out by those who are properly experienced in these analytical procedures. Adequate precision will only be obtained if all the details of the separation and extraction procedures are strictly observed.

2 References

ISO 1042, *Laboratory glassware — One-mark volumetric flasks.*

ISO 1512, *Paints and varnishes — Sampling.*

ISO 1513, *Paints and varnishes — Examination and preparation of samples for testing.*

ISO 3696, *Water for laboratory use — Specifications.*¹⁾

ISO 3856, *Paints and varnishes — Determination of "soluble" metal content.*

ISO 6714, *Paints and varnishes — Preparation of acid extracts from dried films.*¹⁾

3 Definitions

For the purpose of this International Standard the following definitions apply.

3.1 pigment : All particulate matter insoluble in the selected extracting solvent (6.2).

3.2 "soluble" metal content of liquid paint : The metal content of the pigment that is soluble in a defined dilute acid plus the total metal content present in the liquid portion of the paint.

NOTE — The defined dilute acid for the purpose of this International Standard is 0,07 mol/l hydrochloric acid.

3.3 "soluble" metal content of paint in powder form : The metal content of the paint that is soluble in a defined dilute acid (see note to 3.2).

4 Principle

4.1 Dilution of a sample of the liquid product to be tested with a suitable solvent, followed by centrifugal separation of the pigment in the sample. Three methods, dependent on the binder of the product being examined, are described for the separation of the pigment (see 6.4).

4.2 Extraction with 0,07 mol/l hydrochloric acid of the separated pigment at a mass : volume ratio between the pigment and the hydrochloric acid of 1 : 15, as described in 8.2, except for the determination of "soluble" lead when the total lead content of the paint (see 8.3.1.5) equals or exceeds 1 % (*m/m*). In that case, extraction of the separated pigment at a mass : volume ratio between the lead-containing pigment in the sample and the hydrochloric acid of 1 : 1 000, as described in 8.3.

NOTE — For the purpose of this International Standard, it has been assumed that the lead content of a lead-containing pigment is 60 % (*m/m*). This corresponds to the content found in most lead chromes (see 8.3.2.1).

¹⁾ At present at the stage of draft.

ISO 6713-1984 (E)

4.3 Evaporation to dryness of the liquid portion of the product obtained by centrifuging (4.1). Preparation of the residue for analysis by dry ashing and extraction of the ash with nitric acid.

4.4 For paints in powder form, extraction of the product to be tested as in 4.2, without prior separation of the binder (see clause 7).

5 Sampling

Take a representative sample of the product to be tested as described in ISO 1512.

6 Separation of the pigment from the liquid paint sample

6.1 Preparation of the test sample

Examine and prepare the sample for testing as described in ISO 1513. If any skin is present, remove it as far as possible. Thoroughly stir the sample and, if necessary, pass it through a sieve of nominal aperture 150 μm to remove any remaining skin and other extraneous matter.

6.2 Reagents

WARNING — In selecting the solvent to be used, possible toxic and other hazards and necessary precautions should be taken into consideration.

Select a solvent that effects the optimum separation of the pigment. The solvent selected shall be recorded and subsequently reported as required in ISO 3856.

Examples of suitable solvents or solvent mixtures are as follows :

a) for solvent-thinned paints, printing inks and similar products (method A) :

Toluene/ethanol (4 + 1) (for air drying paints)
Xylene/butan-1-ol (9 + 1) (for stoving paints)
Toluene (for chlorinated rubber paints)
Butanone [methyl ethyl ketone, MEK] (for products containing cellulose nitrate)

b) for emulsion paints based on aqueous polymer dispersions (method B) :

Acetone
1,1,1-Trichloroethane
Tetrahydrofuran

c) for plastisols and organosols based on polyvinyl chloride (PVC) and its copolymers and for paints based on other non-aqueous polymer dispersions (method C) :

Tetrahydrofuran
Cyclohexanone
Cyclopentanone

6.3 Apparatus

Ordinary laboratory apparatus and glassware, and in particular :

6.3.1 Suitable laboratory centrifuge, with tubes of inert material of capacity 50 or 100 ml. A centrifuge, capable of imparting a relative centrifugal acceleration of 100 km/s^2 , is preferred.

6.3.2 Air-ventilated oven, capable of being maintained at 105 ± 2 °C.

6.3.3 Stopped glass container, of at least 2 l capacity.

6.4 Procedure

6.4.1 Method A (for solvent-thinned paints, printing inks and similar products)

Weigh, to the nearest 10 mg, a number (see note 1) of centrifuge tubes (6.3.1). Add 10 to 20 g (see note 2) of the prepared sample (6.1) to each tube, taking care to avoid contamination of the walls and lip of the tube. *Immediately* weigh the tubes and contents to the nearest 10 mg. Approximately half fill the tubes with the selected solvent (6.2) and stir thoroughly using a glass rod. Wash each glass rod thoroughly with the solvent, adding all washings to the appropriate tube. Balance the opposing centrifuge tubes to within 0,1 g by adding further solvent, taking care to ensure that an adequate working level is not exceeded. Centrifuge until there is a complete separation into a liquor and a pigment cake. Decant the supernatant liquor from all the tubes comprising a "set" (see note 1) into the container (6.3.3).

Add further solvent to each tube and mix thoroughly as specified above, taking care to disperse the pigment cake completely. Repeat the centrifuging and transfer the liquor to the same container. Repeat the addition of solvent, centrifuging and transfer of liquor for further three times, taking special care, as before, to thoroughly disperse the pigment cake. As a final treatment for the pigment cake and to assist rapid drying, use acetone in place of the selected solvent. Add the acetone and mix, taking special care to disperse the whole pigment cake. Centrifuge and transfer the liquor as before to the container. Retain the container with the combined extracts for the procedure described in clause 9.

After ensuring that excess acetone has evaporated, place the centrifuge tubes in the oven (6.3.2), maintained at 105 ± 2 °C, for a minimum period of 3 h. Remove, transfer to a desiccator, allow to cool to ambient temperature and weigh each tube and contents to the nearest 10 mg. Return the tubes and contents to the oven for a minimum period of 1 h, allow to cool to ambient temperature in the desiccator and reweigh. Repeat the heating, cooling and weighing operations until the results of two consecutive weighings do not differ by more than 10 mg. Calculate the pigment content of the paint as a percentage by mass of the paint sample.

At the end of the separation procedure, check that the dried pigment cake can be crumbled easily to indicate that the binder has been satisfactorily extracted. If the cake remains cohesive, repeat the whole procedure on the original paint using a more suitable solvent or solvent mixture.

NOTES

1 Subsequent acid extractions should be carried out in duplicate and therefore sufficient paint and tubes should be taken so that at least 10 g of pigment is obtained. The number of tubes (usually four) required for each sample is referred to as a "set".

2 The mass of paint taken is dependent upon the capacity of the centrifuge tubes employed and on the expected pigment content of the paint.

6.4.2 Method B (for emulsion paints based on aqueous polymer dispersions)

Carry out the separation as described in 6.4.1, but use acetone for the first and the final treatments and the selected solvent for the five intermediate treatments of the pigment cake.

6.4.3 Method C [for plastisols and organosols based on polyvinyl chloride (PVC) and its copolymers and for paints based on other non-aqueous polymer dispersions].

Carry out the separation as described in 6.4.1, but using a mass of sample such that it may be diluted with solvent in the ratio of 1 : 10. This ratio is necessary to obtain a practical sedimentation rate. Stir very thoroughly for sufficient time to convert the polymer from the dispersed to the dissolved state, using gentle heating if necessary.

6.5 Blank test solution

Prepare a mixture of the solvents using the same proportions as were required for the separation (6.4). Retain the mixture for use as the blank in the determinations described in clause 9.

7 Treatment of paints in powder form

As no prior separation of the pigment from the binder is necessary (see 4.4), extract the test portion (see clause 5) using the procedure described in 8.2 or 8.3. If the material resists dispersion in the hydrochloric acid, add a known volume of a suitable wetting agent.

8 Acid extraction of the separated pigment and of paints in powder form

NOTE — The mass of the pigment to be taken for the extraction may be reduced, for example because of a relatively high "soluble" metal content. In this case, the volume of the extraction liquid should be changed in such a manner that the specified ratio of pigment to extraction liquid is maintained.

8.1 Treatment of the separated pigment

Take all the dried pigment from the tubes in one set obtained by the procedure described in 6.4. Carefully break up the pigment cake by placing it between two sheets of glazed paper and by the application of a minimum pressing or rolling action without any grinding effect disperse it so that all the pigment just passes through a 500 μm sieve (see the note). Ensure that all the pigment passes through the sieve and is collected.

Mix the sieved pigment, place it all in a weighing bottle and transfer to the oven (6.3.2), maintained at 105 ± 2 °C, for 2 h. Store the bottle and contents in a desiccator until required for the extraction procedure (8.2 or 8.3).

NOTE — Excessive force can affect the surface of a pigment and, thereby, produce variable results. If poor reproducibility is experienced or if excessive force is required to break up the pigment cake, the procedure for the extraction and dispersion of the pigment cake should be re-examined.

8.2 Method for the acid extraction of "soluble" metals [including lead when the total lead content of the paint is less than 1 % (*m/m*)] (mass : volume ratio 1 : 15).

8.2.1 Reagents

During the analysis, use only reagents of recognized analytical grade and water of at least grade 3 purity according to ISO 3696.

8.2.1.1 Hydrochloric acid diluted, $c(\text{HCl}) = 0,07$ mol/l.

8.2.1.2 Hydrochloric acid strong, 1 + 1.

Dilute 1 part by volume of hydrochloric acid ($\rho \approx 1,18$ g/ml) with 1 part by volume of water.

8.2.1.3 Ethanol, minimum 95 % (V/V).

8.2.2 Apparatus

Ordinary laboratory apparatus and glassware and, in particular :

8.2.2.1 Suitable mechanical stirrer (see the note in 8.2.3).

8.2.2.2 pH meter and electrodes.

8.2.2.3 Membrane filter, pore diameter 0,15 μm , or other suitable filter capable of giving a clear filtrate in 8.2.3 and 8.3.2.4.

8.2.2.4 Filtration apparatus, for the membrane filter (8.2.2.3).

8.2.2.5 Water-bath, capable of being maintained at 23 ± 2 °C.

8.2.3 Procedure

Carry out the extraction of the prepared pigment (8.1) or of the paint in powder form (clause 7) in duplicate. Protect the test portion from direct sunlight during the extraction and before the analysis.

Weigh $5,0 \pm 0,01$ g of the sample into a clean, dry 150 ml beaker. Wet the test portion [except when it is a paint in powder form (see clause 7)] with 2 ml of the ethanol (8.2.1.3) or the minimum larger quantity to wet the test portion, fit the

ISO 6713-1984 (E)

stirrer (8.2.2.1) and add 75 ml of the dilute hydrochloric acid (8.2.1.1) previously adjusted to 23 ± 2 °C by means of the water-bath (8.2.2.5). Place the beaker in the water-bath and immediately commence stirring the mixture (see the note). Insert the electrodes of the pH meter (8.2.2.2) into the mixture and, if necessary, adjust the pH to that of the dilute hydrochloric acid (8.2.1.1), using the strong hydrochloric acid (8.2.1.2).

Continue stirring for 15 ± 1 min, checking that the temperature of the mixture is maintained at 23 ± 2 °C throughout the test period. Maintain the pH of the mixture by carefully adding the strong hydrochloric acid (8.2.1.2). At the end of the period of stirring, allow the mixture to stand for a further 15 ± 1 min at 23 ± 2 °C. Then decant the mixture through the membrane filter (8.2.2.3) using the filtration apparatus (8.2.2.4) and collect the filtrate obtained in the first 10 min (which should be a clear solution) in a suitable glass container. Immediately stopper the container.

Retain the filtered extract for the determination of the various "soluble" metals as described in the appropriate parts of ISO 3856. Take appropriate aliquot portions for each determination.

Carry out the determination, as described in the appropriate parts of ISO 3856, of the "soluble" metal content of the filtrate as soon as possible and within 4 h of the preparation of the extract.

NOTE — During the whole period of extraction, the speed of the stirrer should be adjusted so that the pigment is kept in continuous suspension whilst taking care to avoid splashing.

8.3 Method for the acid extraction of "soluble" lead [when the total lead content of the paint equals or exceeds 1 % (m/m)] (mass : volume ratio 1 : 1 000)

8.3.1 Preliminary determination of total lead

NOTE — In order to be able to maintain constant the ratio of the mass of lead-containing sample to the volume of the hydrochloric acid extractant, it is necessary to determine first the total lead content of the prepared pigment (8.1) or paint in powder form (clause 7). From the total lead content, it is possible to calculate (8.3.2.1) the mass of sample to be used in the extraction.

8.3.1.1 Principle

Dissolution of the prepared pigment or paint in powder form in nitric acid and hydrogen peroxide. Determination of the lead content by flame atomic absorption spectrometry as described in ISO 3856/1.

8.3.1.2 Reagents

During the analysis, use only reagents of recognized analytical grade and water of at least grade 3 purity according to ISO 3696.

8.3.1.2.1 Nitric acid, $\rho \approx 1,40$ g/ml, about 65 % (m/m).

8.3.1.2.2 Hydrogen peroxide, approximately 30 % (m/m) solution.

8.3.1.2.3 Hydrochloric acid, $c(\text{HCl}) = 0,07$ mol/l.

8.3.1.3 Apparatus

Ordinary laboratory apparatus and :

Hot-plate, with energy regulation control.

8.3.1.4 Procedure

8.3.1.4.1 Extract solution

Carry out the procedure on the prepared pigment (8.1) or paint in powder form (clause 7) in duplicate.

Weigh, to the nearest 1 mg, 0,1 g of the sample and place in a 150 ml conical flask. Add 10 ml of the nitric acid (8.3.1.2.1) followed by two drops of the hydrogen peroxide solution (8.3.1.2.2). Swirl to mix and boil gently on the hot-plate (8.3.1.3) for 5 min, taking care not to boil dry (see the note). Allow to cool slightly and rinse the sides of the flask with approximately 10 ml of water. Boil for a further 5 min. Allow to cool, filter if necessary, and quantitatively transfer the contents of the flask into a 250 ml one-mark volumetric flask. Make up to the mark with water, stopper and mix well. This is the extract solution.

NOTE — Paints in powder form may require a more drastic treatment to dissolve the lead-containing pigments. For these products, it may be necessary to add more nitric acid and hydrogen peroxide solution and to increase the heating time.

8.3.1.4.2 Preparation of test solution

From each extract solution (8.3.1.4.1) take an aliquot portion, of size determined by the expected lead content in the sample (see the table). Transfer the aliquot portion into a 100 ml one-mark volumetric flask, make up to the mark with the hydrochloric acid (8.3.1.2.3) and mix well.

Table

Expected lead content	Aliquot portion
% (m/m)	ml
less than 2	50
2 up to 10	25
10 up to 90	10

8.3.1.4.3 Preparation of reagent blank

Repeat the procedures described in 8.3.1.4.1 but omitting the sample. Take an aliquot portion of the blank solution equal to that of the extract solution used in preparing the test solution (8.3.1.4.2).

8.3.1.4.4 Determination

Use the test solutions and the reagent blank solutions obtained in 8.3.1.4.2 and 8.3.1.4.3 respectively as the test and blank solutions, described in ISO 3856/1, sub-clause 3.4.2.3. Determine their lead content by the flame atomic absorption spectrometric method described in ISO 3856/1 in duplicate.

If the duplicate readings obtained differ by more than 2 % absolute, prepare further fresh test solutions (8.3.1.4.2) and repeat the procedures.

8.3.1.5 Calculation

Calculate the total lead content, using the equation

$$T = \frac{2,5 c}{m_0 \times V}$$

where

c is the lead concentration, in micrograms per millilitre, of the test solution obtained from the calibration graph, corrected for the blank;

m_0 is the mass, in grams, of the test portion (8.3.1.4.1);

T is the total lead content of the prepared pigment or paint in powder form, as a percentage by mass;

V is the volume, in millilitres, of the aliquot portion of the extract, taken in 8.3.1.4.2;

Calculate the mean of the two results.

8.3.2 Extraction procedure

8.3.2.1 Calculation of mass of test portion

a) For prepared pigments

From the pigment content of the liquid paint (see clause 6), together with the lead content of the prepared pigment, T , calculate the total lead content of the paint. If the total lead content equals or exceeds 1 % (m/m) of the paint, calculate m_1 as described below and carry out the extraction procedure described in 8.3.2.4.

b) For paints in powder form

If the total lead content of the paint in powder form equals or exceeds 1 % (m/m), calculate m_1 as described below and carry out the extraction procedure described in 8.3.2.4.

From the total lead content of the prepared pigment or of the paint in powder form (as calculated in 8.3.1.5), calculate the mass of the test portion for the determination of the "soluble" lead content, using the equation

$$m_1 = \frac{60}{T} \times 0,5 = \frac{30}{T}$$

where

m_1 is the mass, in grams, of the test portion;

T is the total lead content of the prepared pigment or paint in powder form, as a percentage by mass;

60 is the average lead content, as a percentage by mass, of typical lead chrome pigments (see the note in 4.2).

8.3.2.2 Reagents

See 8.2.1.

8.3.2.3 Apparatus

See 8.2.2.

8.3.2.4 Procedure

Carry out the extraction of the prepared pigment (8.1) or of the paint in powder form (clause 7) in duplicate. Protect the test portion from direct sunlight during the extraction and before the analysis.

Weigh the test portion (m_1) to the nearest 1 mg and place it in a clean, dry 1 000 ml beaker. Wet the test portion [except when it is a paint in powder form (see clause 7)] with 2 ml of the ethanol (8.2.1.3), or the minimum larger quantity to wet the test portion, fit the stirrer (8.2.2.1) and add 500 ml of the dilute hydrochloric acid (8.2.1.1), previously adjusted to 23 ± 2 °C by means of the water-bath (8.2.2.5). Place the beaker in the water-bath and immediately commence stirring the mixture (see the note). Insert the electrodes of the pH meter (8.2.2.2) into the mixture and, if necessary, adjust the pH to that of the dilute hydrochloric acid (8.2.1.1), by adding the strong hydrochloric acid (8.2.1.2).

Continue stirring for 60 ± 1 min, checking that the temperature of the mixture is maintained at 23 ± 2 °C throughout the test period. Maintain the pH of the mixture by carefully adding the strong hydrochloric acid (8.2.1.2). At the end of the period of stirring, allow the mixture to stand for a further 60 ± 1 min at 23 ± 2 °C. Then decant the mixture through the membrane filter (8.2.2.3) using the filtration apparatus (8.2.2.4) and collect the filtrate obtained in the first 10 min (which should be a clear solution) in a suitable glass container. Immediately stopper the container.

Retain the filtered extract for the determination of the "soluble" lead as described in ISO 3856/1. Take appropriate aliquot portions for each determination.

Carry out the determination of the "soluble" lead content of the filtrate as soon as possible and within 4 h of the preparation of the extract.

NOTE — During the whole period of extraction, the speed of the stirrer should be adjusted so that the pigment is kept in continuous suspension whilst taking care to avoid splashing.

8.4 Blank test solution

Take 75 or 500 ml, as appropriate, of the dilute hydrochloric acid (8.2.1.1) and, if necessary, add 2 ml of the ethanol (8.2.1.3) (see 8.2.3 and 8.3.2.4). Retain this solution for the blank determinations on the pigment portion of the paint as described in the appropriate parts of ISO 3856.

9 Treatment of the extracted liquid portion

9.1 Reagents

During the analysis, use only reagents of recognized analytical grade and only water of at least grade 3 purity according to ISO 3696.

9.1.1 Nitric acid 1 + 1.

Dilute 1 part by volume of nitric acid, 65 % (m/m) ($\rho \approx 1,40$ g/ml) with 1 part by volume of water.

ISO 6713-1984 (E)

9.1.2 Hydrochloric acid diluted, $c(\text{HCl}) = 0,07 \text{ mol/l}$.

Use the same acid as in 8.2.1.1.

9.2 Apparatus

9.2.1 Water-bath.

9.2.2 Hot-plate.

9.2.3 Muffle furnace, capable of being maintained at $475 \pm 25 \text{ }^\circ\text{C}$.

9.2.4 One-mark volumetric flask, of capacity 100 ml, complying with the requirements of ISO 1042.

9.3 Procedure

Carry out the following procedure in duplicate on the extracted liquid portion.

Carry out the same procedure on the blank test solution (6.5) using the same quantities of all reagents.

Transfer the contents of the stoppered glass container, obtained according to 6.4, into a suitable graduated vessel and dilute to a specific volume with the solvent or solvent mixture (6.2). Transfer an aliquot portion (see note 6) to a porcelain or silica dish of suitable capacity. Evaporate (see note 1) the main portion of the solvent, using the water-bath (9.2.1) (see note 2).

Place the dish on the hot-plate (9.2.2) and slowly increase the temperature in order to remove all residual solvent (see note 3). Gradually increase the temperature of the hot-plate until the material begins to char. Then transfer the dish to the muffle furnace (9.2.3), maintained at $475 \pm 25 \text{ }^\circ\text{C}$, and ash (see note 4). When the ashing is complete, remove the dish from the muffle furnace and allow it to cool to ambient temperature. Break up the ash into fine particles with a glass rod and leave the rod in the dish during the following filtration step (see note 5).

Taking care to avoid loss of material if the ash reacts vigorously, slowly add 10 ml of the nitric acid (9.1.1). Heat carefully on the hot-plate until 2 to 3 ml of solution remain. Add an additional 10 ml of the nitric acid and continue heating on the hot-plate until less than 5 ml of solution remain. Add 20 to 25 ml of water and filter the solution through a medium porosity filter paper into the 100 ml one-mark volumetric flask (9.2.4). If the filtrate is not clear, refilter through fine porosity filter paper.

Wash the dish and the filter paper several times with water and add the washings to the flask. Dilute to the mark with the hydrochloric acid (9.1.2) and mix well.

Retain the extract for the determination of the various "soluble" metals as described in the appropriate parts of ISO 3856. Take appropriate aliquot portions for each determination.

NOTES

1 If the paint sample contains cellulose nitrate, add 2 g of liquid paraffin to the liquid portion of the paint before removing the volatile solvents.

2 If desired, the bulk of the solvent may be removed by distillation from a flask. This latter method is preferred when, for example, the solvents include butanol or xylene.

3 If the paint sample contains chlorinated compounds, acidic products will be formed during ashing. Therefore, before ashing, the residue obtained on evaporation to dryness of the extract of the liquid portion should be covered with anhydrous sodium carbonate to neutralize these products. Approximately 1 g of sodium carbonate should be added for each 1 g of residue. If it should be necessary to add a relatively large quantity of sodium carbonate, it is desirable to take an appropriate aliquot portion of the combined extract (6.4).

The same procedure should also be applied to the blank solution.

4 Ashing at above $500 \text{ }^\circ\text{C}$ may result in the loss of some lead and cadmium by volatilization.

5 If the paint sample contains both antimony and lead, there is the likelihood of the formation of an insoluble compound that will be retained in the acid-insoluble residue; consequently low results will be obtained. To avoid these low results, ash the samples as directed, grind the ash to a fine powder, replace it in the same container and mix it with approximately six times its own mass of a mixture of equal parts of anhydrous sodium carbonate and sulfur. Cover the container and heat over a moderate flame until there is no odour of sulfur dioxide. This should take 1 to 2 h.

Cool and digest with a small quantity of hot water until the melt is completely broken up. Filter, transfer the residue to the filter paper with a 1 % (*m/m*) sodium sulfide solution and wash the residue with the same solution. Discard the filtrate and washings (if only lead is to be determined) or reserve the filtrate and washings for the antimony determination (if both lead and antimony are to be determined).

Dissolve the residue in the minimum quantity of boiling nitric acid, $c(\text{HNO}_3) \approx 5 \text{ mol/l}$; filter and wash with hot water into a 100 ml one-mark volumetric flask (9.2.4). Dilute to the mark with the dilute hydrochloric acid (9.1.2) and mix well.

The same procedure should also be applied to the blank solution.

6 When calculating the "soluble" metal content, as described in the appropriate parts of ISO 3856, it will be necessary to take account of the total volume of the extracted liquid portion.