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Steel and iron — Review of available methods of analysis

Aciers et fontes — Vue d'ensemble des méthodes d'analyse disponibles

Reference number
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Foreword

ISO (the International Organization for Standardization) is a worldwide federation of national standards bodies (ISO member bodies). The work of preparing International Standards is normally carried out through ISO technical committees. Each member body interested in a subject for which a technical committee has been established has the right to be represented on that committee. International organizations, governmental and non-governmental, in liaison with ISO, also take part in the work. ISO collaborates closely with the International Electrotechnical Commission (IEC) on all matters of electrotechnical standardization.

The main task of technical committees is to prepare International Standards, but in exceptional circumstances a technical committee may propose the publication of a Technical Report of one of the following types:

— type 1, when the required support cannot be obtained for the publication of an International Standard, despite repeated efforts;

— type 2, when the subject is still under technical development or where for any other reason there is the future but not immediate possibility of an agreement on an International Standard;

— type 3, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example).

Technical Reports of types 1 and 2 are subject to review within three years of publication, to decide whether they can be transformed into International Standards. Technical Reports of type 3 do not necessarily have to be reviewed until the data they provide are considered to be no longer valid or useful.

ISO/TR 9769, which is a Technical Report of type 3, was prepared by Technical Committee ISO/TC 17, *Steel*.

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The main task of ISO technical committees is to prepare International Standards. In exceptional circumstances a technical committee may propose the publication of a technical report of one of the following types:

- type 1, when the necessary support within the technical committee cannot be obtained for the publication of an International Standard, despite repeated efforts;

- type 2, when the subject is still under technical development, requiring wider exposure;

- type 3, when a technical committee has collected data of a different kind from that which is normally published as an International Standard ("state of the art", for example).

Technical reports are accepted for publication directly by ISO Council. Technical reports types 1 and 2 are subject to review within three years of publication, to decide if they can be transformed into International Standards. Technical reports type 3 do not necessarily have to be reviewed until the data they provide are considered to be no longer valid or useful.

ISO/TR 9769 was prepared by Technical Committee ISO/TC 17, Steel.

It was decided to publish this document in the form of a technical report type 3.

This technical report was revised in accordance with Resolution No.1/88 taken at the 16th Plenary Meeting of ISO/TC 17 held in 1988-10-10/14.

1 Scope

This Technical Report aims to facilitate reference to the available international standard method(s) for the determination of required element(s) in steel and iron.

In this Technical Report, field of application, method of determination (principle) and precision (see informative annex A) of each standard are stated.

2 List of International Standards

2.1 Al Aluminium

Reference	Ed	Pages	Title
ISO 9658: 1990	1	11	Steel - Determination of aluminium content - Flame atomic absorption spectrometric method

2.2 As Arsenic

to be prepared.

2.3 B Boron

ISO 10153: 1991	1	10	Steel - Determination of boron content - Curcumin spectrophotometric method

2.4 Bi Bismuth

to be prepared.

2.5 C Carbon

ISO 437:1982	1	4	Steel and cast iron - Determination of total carbon content - Combustion gravimetric method
ISO/TR 4830-4 :1978	1	8	Steel - Determination of low carbon contents - Part 4: Coulometric method after combustion
ISO 9556:1989	1	8	Steel and iron - Determination of total carbon content - Infrared absorption method after combustion in an induction furnace

2.6 Ca Calcium

ISO/DIS 10697-1			Steel - Ditermination of calcium content by flame atomic absorption spectrometry - Part 1: Determination of acid-soluble calcium content

2.7 Ce Cerium

to be prepared.

2.8 Co Cobalt

under preparation.

2.9 Cr Chromium

Reference	Ed	Pages	Title
ISO 4936:1984	1	3	Steel and cast iron - Determination of chromium content - Diphenylcarbazide spectrophotometric method
ISO 4937:1986	1	8	Steel and iron - Determination of chromium content - Potentiometric or visual titration method
ISO/DIS 10138			Steel and iron - Determination of chromium content - Flame atomic absorption spectrometric method

2.10 Cu Copper

ISO 4943:1985	1	6	Steel and cast iron - Determination of copper content - Flame atomic absorption spectrometric method
ISO 4946:1984 (Corrected and reprinted:1986)	1	6	Steel and cast iron - Determination of copper content - 2,2'-Diquinolyl spectrophotometric method

2.11 Mg Magnesium

to be prepared.

2.12 Mn Manganese

ISO 629:1982	1	4	Steel and cast iron - Determination of manganese content - Spectrophotometric method
ISO/TR 10281 :1990	1	10	Steel and iron - Determination of manganese content - Flame atomic absorption spectrometric method

2.13 Mo Molybdenum

Reference	Ed	Pages	Title
ISO 4941:1978	1	4	Steels and cast irons - Determination of molybdenum content - Photometric method

2.14 N Nitrogen

ISO 4945:1977	1	6	Steel - Determination of nitrogen content - Spectrophotometric method

2.15 Nb Niobium

ISO 9441:1988	1	6	Steel - Determination of niobium content - PAR spectrophotometric method

2.16 Ni Nickel

ISO 4938:1988	1	8	Steel and iron - Determination of nickel content - Gravimetric or titrimetric method
ISO 4939:1984 (Corrected and reprinted:1986)	1	6	Steel and cast iron - Determination of nickel content - Dimethylglyoxime spectrophotometric method
ISO 4940:1985	1	7	Steel and cast iron - Determination of nickel content - Flame atomic absorption spectrometric method

2.17 O Oxygen

to be prepared.

2.18 P Phosphorus

ISO 2732:1984	2	3	Steel and cast iron - Determination of phosphorus content - Phosphovanadomolybdate spectrophotometric method

2.19 Pb Lead

to be prepared.

2.20 S Sulfur

Reference	Ed	Pages	Title
ISO 671:1982	1	5	Steel and cast iron - Determination of sulfur content - Combustion titrimetric method
ISO 4934:1980	1	5	Steel and cast iron - Determination of sulfur content - Gravimetric method
ISO 4935:1989	1	7	Steel and iron - Determination of sulfur content - Infrared absorption method after combustion in an induction furnace

2.21 Sb Antimony

under preparation.

2.22 Si Silicon

ISO 439:1982	1	3	Steel and cast iron - Determination of total silicon - Gravimetric method
ISO 4829-1:1986	1	7	Steel and cast iron - Determination of total silicon content - Reduced molybdosilicate spectrophotometric method - Part 1: Silicon contents between 0,05 and 1,0 %
ISO 4829-2:1988	1	5	Steel and iron - Determination of total silicon content - Reduced molybdosilicate spectrophotometric method - Part 2: Silicon contents between 0,01 and 0,05 %

2.23 Sn Tin

to be prepared.

2.24 Ta Tantalum

to be prepared.

2.25 Ti Titanium

ISO/DIS 10280			Steel and iron - Determination of titanium content - Diantipyrylmethane spectrophotometric method

2.26 V Vanadium

Reference	Ed	Pages	Title
ISO 4942:1988	1	6	Steel and iron - Determination of vanadium content - N-BPHA spectrophotometric method
ISO 4947:1986	1	6	Steel and cast iron - Determination of vanadium content - Potentiometric titration method
ISO 9647:1989	1	8	Steel and iron - Determination of vanadium content - Flame atomic absorption spectrometric method

2.27 W Tungsten

to be prepared.

2.28 Zr Zirconium

to be prepared.

3 Synopsis of method

3.1 Al Aluminium

3.1.1 ISO 9658: 1990 Flame atomic absorption spectrometric method

(1) Scope

Determination of acid-soluble and/or total aluminium contents between 0,005 % (m/m) and 0,20 % (m/m) in non-alloyed steel.

(2) Definition

Acid-soluble aluminium for the method is defined as the aluminium dissolved in the hydrochloric and nitric acids mixture as specified in the procedure.

(3) Principle

Dissolution of a test portion in dilute hydrochloric and nitric acids.

Fusion of the acid-insoluble material with orthoboric acid-potassium carbonate mixture.

Spraying of the solution into a dinitrogen monoxide acetylene flame.

Spectrometric measurement of the atomic absorption of the 309,3 nm spectral line emitted by an aluminium hollow cathode lamp.

(4) Precision: see informative annex A.

Figure A.1.1; Precision for the determination of acid-soluble aluminium content
Figure A.1.2; Precision for the determination of total aluminium content

3.2 As Arsenic

to be prepared.

3.3 B Boron

3.3.1 ISO 10153: 1991 Curcumin spectrophotometric method

(1) Scope

Determination of boron contents between 0,000 5 % (m/m) and 0,012 % (m/m) in steel.

(2) Principle

Dissolution of a test portion in hydrochloric and nitric acids.

Decomposition of boron compounds (nitrides etc.) with orthophosphoric and sulfuric acids at a temperature of 290°C.

Formation of a coloured complex between orthoboric acid and curcumin in buffered acetic acid medium.

Spectrophotometric measurement at a wavelength of about 543 nm.

(3) Precision: see information annex A.

Figure A.3.1; Precision for the determination of boron content by ISO 10153

3.4 Bi Bismuth

to be prepared.

3.5 C Carbon

3.5.1 ISO 437: 1982, Combustion gravimetric method

(1) Scope

Determination of total carbon contents not less than 0,1 % (m/m) in steel and cast iron.

(2) Principle

Combustion of a test portion at a high temperature (1 200°C to 1 350°C) in a current of pure oxygen, if necessary in the presence of a flux and transformation of carbon into carbon dioxide.

Absorption of the carbon dioxide carried by current of oxygen in soda asbestos contained in a weighed absorption bulb, and determination of the increase in mass.

3.5.2 ISO/TR 4830-4: 1978, Part 4: Coulometric method after combustion

(1) Scope

Determination of carbon contents between 0,005 % (m/m) and 0,1 % (m/m) in steels.

(2) Principle

Combustion of a test portion at a high temperature (> 1 250°C; see note below) in a current of pure oxygen, converting the carbon to carbon dioxide.

Entrainment of the carbon dioxide by the current of oxygen, and separation from the other volatile oxides.

Absorption of the carbon dioxide in a barium hydroxide solution containing barium perchlorate: during the absorption, re-establishment of the initial barium hydroxide concentration by electrolysis of the barium perchlorate, the variation in the pH of the solution being followed by a pH meter.

Measurement of the quantity of electricity required for re-establishment of the initial pH, in order to deduce the quantity of barium hydroxide formed and hence the quantity of carbon dioxide neutralized.

The pH of the absorbing solution must always be equal to or greater than 9,0.

NOTE - Certain types of apparatus are incapable of obtaining the temperature of 1 250°C necessary to comply with the requirements of this Technical Report.

3.5.3 ISO 9556: 1989, Infrared absorption method after combustion in an induction furnace

(1) Scope

Determination of total carbon contents between 0,003 % (m/m) and 4,5 % (m/m) in steel and iron.

(2) Principle

Combustion of a test portion with accelerator at a high temperature in an induction furnace in a current of pure oxygen and transformation of carbon into carbon dioxide and/or carbon monoxide.

Measurement by infrared absorption of the carbon dioxide and/or carbon monoxide carried by a current of oxygen.

(3) Precision: see informative annex A.

Figure A.5.1; Precision for the determination of total carbon content by ISO 9556

3.6 Ca Calcium

3.6.1 ISO/DIS 10697-1:Flame atomic absorption spectrometric method - Part 1: Determination of acid-soluble calcium content

(1) Scope

Determination of acid soluble calcium contents between 0,0005 % (m/m) and 0,003 % (m/m) in steel.

(2) Principle

Dissolution of a test portion in hydrochloric and nitric acids.

Addition of potassium chloride-lanthanum nitrate solution as a spectrochemical buffer.

Spraying of the solution into a dinitrogen monoxide-acetylene flame.

Spectrometric measurement of the atomic absorption of the 422,7 nm spectral line emitted by a calcium hollow cathode lamp.

(3) Precision: see informative annex A.

Figure A.6.1; Precision for the determination of acid-soluble calcium content by ISO/DIS 10697-1.

3.7 Ce Cerium

to be prepared.

3.8 Co Cobalt

under preparation.

3.9 Cr Chromium

3.9.1 ISO 4936: 1984, Diphenylcarbazide spectrophotometric method

(1) Scope

Determination of chromium contents between 0,01 % (m/m) and 0,25 % (m/m) in steel and cast iron.

(2) Principle

Dissolution of a test portion in nitric and perchloric acids.

Oxidation of chromium in a phosphoric perchloric medium by potassium permanganate.

Oxidation of diphenylcarbazide by chromium(VI).

Spectrophotometric measurement of the oxidized form of diphenylcarbazide at a wavelength of about 540 nm.

3.9.2 ISO 4937: 1986, Potentiometric or visual titration method

(1) Scope

Determination of chromium contents between 0,25 % (m/m) and 35 % (m/m) in steel and iron. (If vanadium is present, the visual titration is applicable only to test portions containing less than 3 mg of vanadium.)

(2) Principle

Dissolution of a test solution with appropriate acids.

Oxidation of chromium in an acid medium to chromium(VI) by ammonium peroxydisulfate in the presence of silver sulfate.

Reduction of manganese(VII) by hydrochloric acid.

Reduction of chromium(VI) by ammonium iron(II) sulfate standard solution.

In the case of potentiometric detection, determination of the equivalence point by measurement of the potential variation when the ammonium iron(II) sulfate standard solution is being added.

In the case of visual detection, titration of the excess ammonium iron(II) sulfate by potassium permanganate standard solution which also acts as the indicator.

(3) Precision: see informative annex A.

Figures A.9.1 and A.9.2;	Precision for the determination of chromium content by ISO 4937

3.9.3 ISO/DIS 10138: Flame atomic absoroption spectrometric method

(1) Scope

Determination of chromium contents between 0,002 % (m/m) and 2,0 % (m/m) in non-alloy and low-alloy steels and iron.

(2) Principle

Dissolution of a test portion in hydrochloric acid, followed by oxidation with nitric acid.

Filtration and ignition of the acid insoluble residue
Removal of silica with hydrofluoric acid.

Fusion of the residue with potassium hydrogen sulfate, extraction of the melt in acid and addition of the extract to the reserved filtrate.

Spraying the solution into a dinitrogen monoxide-acetylene flame.

Spectrometric measurement of the atomic absorption of the 357,9 nm or 425,4 nm spectral line emitted by a chromium hollow cathode lamp.

(3) Precision: see informative annex A.

Figure A.9.3; Precision for the determination of chromium content by ISO/DIS 10138

3.10 Cu Copper

3.10.1 ISO 4943: 1985, Flame atomic absorption spectrometric method

(1) Scope

Determination of copper contents between 0,004 % (m/m) and 0,5 % (m/m) in steel and cast iron.

(2) Principle

Dissolution of a test portion in a mixture of hydrochloric, nitric and perchloric acids.

Spraying of the solution into an air-acetylene flame.

Spectrometric measurement of the atomic absorption of the 324,7 nm spectral line emitted by a copper hollow cathode lamp.

(3) Precision: see informative annex A.

Figure A.10.1; Precision for determination of copper content by ISO 4943

3.10.2 ISO 4946: 1984, 2,2'-Diquinolyl spectrophotometric method

(1) Scope

Determination of copper contents between 0,02 % (m/m) and 5 % (m/m) in steel and cast iron.

(2) Principle

Dissolution of a test portion with appropriate acids.

Fuming with perchloric acid to remove hydrochloric and nitric acids and dehydrate silicic acid.

Reduction of copper(II) to copper(I) in hydrochloric acid solution by means of ascorbic acid.

Formation of a coloured compound of copper(I) with 2,2'-diquinolyl.

Spectrophotometric measurement at a wavelength of about 545 nm.

(3) Precision: see informative annex A.

Figure A.10.2; Precision for determination of copper content by ISO 4946

3.11 Mg Magnesium

to be prepared.

3.12 Mn Manganese

3.12.1 ISO 629: 1982, Spectrophotometric method

(1) Scope

Determination of manganese contents between 0,01 % (m/m) and 4 % (m/m) in steel and cast iron.

(2) Principle

Dissolution of a test portion in sulfuric-phosphoric acid mixture and nitric acid.

Treatment of the test solution with perchloric acid.

Formation of the permanganate ion by sodium periodate.

Spectrophotometric measurement of the test solution at wavelength of about 545 nm.

3.12.2 ISO/TR 10281: Flame atomic absoroption spectrometric method

(1) Scope

Determination of manganese contents between 0,002 % (m/m) and 3,0 % (m/m) in non-alloy and low-alloy steels and iron.

(2) Principle

Dissolution of a test portion with hydrochloric acid followed by oxidation with nitric acid.

Ignition of the residue and removal of silica with hydrofluoric acid.

Fusion of the residue with potassium hydrogen sulfate.

Determination of the manganese by means of the spectrometric measurement of the atomic absoroption of the 403,1 nm line emitted by a manganese hollow cathode lamp when the solution is sprayed into an air acetylene flame.

For low levels of manganese the more sensitive line of 279,5 nm may be used.

(3) Precision

Figure A.12.1; Precision for determination of manganese content by ISO/TR 10281

3.13 Mo Molybdenum

3.13.1 ISO 4941: 1978, Photometric method

(1) Scope

Determination of molybdenum contents between 0,003 % (m/m) and 9 % (m/m) in steel and cast iron.

Vanadium and tungsten interfere with the measurement if, because of their contents, the V/Mo ratio is greater than 16 or the W/Mo ratio is greater than 8.

NOTE - Greater V/Mo or W/Mo ratios (up to 300) may, however, be permitted, but in such cases it is necessary to carry out the measurement very quickly after the extraction.

(2) Principle

Dissolution of a test portion in an appropriate mixture of acids and decomposition of the carbides by oxidation.

Quantitative formation of a coloured compound of molybdenum, in the presence of thiocyanate, iron(II) and/or copper(II) ions and extraction of this compound using n-butyl acetate.

Photometric measurement of the coloured compound at a wavelength of about 470 nm.

NOTE - When the conditions of the procedure are respected, the coefficient of molecular absorption is 18 930 ± 60.

3.14 N Nitrogen

3.14.1 ISO 4945: 1977, Spectrophotometric method

(1) Scope

Determination of nitrogen contents between 0,002 % (m/m) and 0,050 % (m/m) in non-alloy and low-alloy steels.

This method allows the determination only of the nitrogen content which can be converted to an ammonium salt and is applicable to non- alloy and low-alloy steels less than 0,6 % (m/m) of silicon.

(2) Principle

Dissolution of a test portion in dilute sulfuric acid.

After concentration, progressive increasing of the temperature to above 300°C.

Separation of ammonia from the ammonium salt formed, by displacement and distillation in a boiling sodium hydroxide medium and collecting in an acid medium.

At ambient temperature, formation of a blue-colored complex between the ammonium ions and phenol in the presence of sodium hypochlorite and sodium pentacyanonitrosylferrate(II) (sodium nitroprusside).

Spectrophotometric measurement of the complex at a wavelength of about 640 nm.

3.15 Nb Niobium

3.15.1 ISO 9441: 1988, PAR spectrophotometric method

(1) Scope

Determination of niobium contents between 0,005 % (m/m) and 1,3 % (m/m) in steel.

(2) Principle

Dissolution of a test portion in hydrochloric acid followed by oxidation with hydrogen peroxide.

Precipitation of niobium and tantalum with phenylarsonic acid, using zirconium as a carrier.

Formation of a complex of niobium with 4-(2-pyridylazo)-resorcinol (PAR) in a sodium tartrate medium buffered by sodium acetate solution adjusted to pH 6,3.

Spectrophotometric measurement of the coloured compound at a wavelength of about 550 nm.

(3) Precision: see informative annex A.

Figure A.15.1; Precision for determination of niobium content by ISO 9441

3.16 Ni Nickel

3.16.1 ISO 4938: 1988, Gravimetric or titrimetric method

(1) Scope

Determination of nickel contents between 0,5 % (m/m) and 30 % (m/m) in steel and iron.

(2) Principle

Dissolution of a test portion with appropriate acids.

Precipitation of the nickel as nickel dimethylglyoxime.

- Cobalt, if present, is oxidized by potassium hexacyanoferrate(III);
- Copper, if present with cobalt, preferably is removed by controlled-potential electrolysis.

Acid dissolution of the precipitate and filtration of the solution, followed by a second precipitation of the nickel as nickel dimethylglyoxime.

In the case of the gravimetric finish, weighing of the dried nickel dimethylglyoxime precipitate.

In the case of the titrimetric finish, acid dissolution of the precipitate, addition of excess EDTA.Na_2 solution and back titration of the excess EDTA.Na_2 by zinc solution using xylenol orange as an indicator.

(3) Precision: see informative annex A.

Figure A.16.1; Precision for determination of nickel content by ISO 4938 - Gravimetric method
Figure A.16.2; Precision for determination of nickel content by ISO 4938 - Titrimetric method

3.16.2 ISO 4939: 1984, Dimethylglyoxime spectrophotometric method

(1) Scope

Determination of nickel contents between 0,10 % (m/m) and 4 % (m/m) in steel and cast iron.

Cobalt, copper and manganese may cause interferences.

(2) Principle

Dissolution of a test portion in hydrochloric, nitric and perchloric acids.

Formation of a coloured complex of nickel(III) with dimethylglyoxime in ammoniacal solution containing iodine and potassium iodide.

Spectrophotometric measurement at a wavelength of about 535 nm.

(3) Precision: see informative annex A.

Figure A.16.3; Precision for determination of nickel content by ISO 4939

3.16.3 ISO 4940: 1985, Flame atomic absorption spectrometric method

(1) Scope

Determination of nickel contents between 0,002 % (m/m) and 0,5 % (m/m) in steel and cast iron.

(2) Principle

Dissolution of a test portion in a mixture of appropriate acids followed by evaporation to fuming with perchloric acid.

Spraying of the solution into an air-acetylene flame.

Spectrometric measurement of the atomic absorption of the 352,5 nm spectral line emitted by a nickel hollow cathode lamp.

NOTE - With some instruments it is not possible to obtain sufficient sensitivity at the wavelength of 352,5 nm for low concentrations of nickel near the bottom end of the application range and in such cases the alternative wavelength of 232,0 nm must be used.

At the wavelength of 352,5 nm, the signal-to-noise ratio is higher than at a wavelength of 232,0 nm. In general, use of the 352,5 nm line will give better reproducibility.

(3) Precision: see informative annex A.

Figure A.16.3; Precision for determination of nickel content by ISO 4940

3.17 O Oxygen

to be prepared.

3.18 P Phosphorus

3.18.1 ISO 2732: 1984, Phosphovanadomolybdate spectrophotometric method

(1) Scope

Determination of phosphorus contents between 0,005 % (m/m) and 1,5 % (m/m) in steel and cast iron, provided that tungsten, niobium, tantalum and zirconium contents are not higher than 1 % (m/m) for each of these four elements and titanium content is not higher than 2 % (m/m).

(2) Principle

Dissolution of a test portion in an oxidizing acid mixture.

Conversion of phosphorus to phosphovanadomolybdate in perchloric-nitric acids solution.

Extraction of phosphovanadomolybdate into 4-methyl-2-pentanone with citric acid present to complex arsenic.

Spectrophotometric measurement at a wavelength of about 425 nm.

3.19 Pb Lead

to be prepared.

3.20 S Sulfur

3.20.1 ISO 671: 1982, Combustion titrimetric method

(1) Scope

Determination of sulfur content in steel and cast iron.

(2) Principle

Combustion of a test portion at a high temperature (1 450°C) in a current of pure oxygen, if necessary in the presence of a metal flux and transformation of sulfur into sulfur dioxide.

Absorption of the gases resulting from combustion, freed from oxide dust, by a solution of potassium sulfate and hydrogen peroxide.

Titration of the absorbent solution with a standard sodium borate solution.

3.20.2 ISO 4934: 1980, Gravimetric method

(1) Scope

Determination of sulfur contents greater than 0,003 % (m/m) in steel and cast iron, excluding steel containing selenium.

The method is particularly suitable as a reference method for the standardization of samples on which certified standard values are to be established.

(2) Principle

Dissolution of a test portion in dilute nitric acid in the presence of bromine, or in dilute nitric acid and concentrated hydrochloric acid in the presence of bromine (with the aid of an appropriate device to prevent sulfur losses).

Addition of hydrofluoric acid and perchloric acid and evaporation of the solution until white fumes of perchloric acid are evolved.

If necessary, volatilization of chromium as chromyl chloride.

Chromatographic separation of the sulfate ions in an alumina column and elution with ammonium hydroxide solution.

Precipitation of the sulfate ions as barium sulfate under carefully controlled conditions and filtering, washing, heating and weighing.

3.20.3 ISO 4935: 1989, Infrared absorption method after combustion in an induction furnace

(1) Scope

Determination of sulfur contents between 0,002 % (m/m) and 0,10 % (m/m) in steel and iron.

(2) Principle

Combustion of a test portion with accelerator at a high temperature in a high frequency induction furnace in a current of pure oxygen and transformation of sulfur into sulfur dioxide.

Measurement by infrared absorption of the sulfur dioxide carried by a current of oxygen

(3) Precision: see informative annex A.

Figure A.20.1; Precision for determination of sulfur content by ISO 4935

3.21 **Sb** Antimony

under preparation.

3.22 Si Silicon

3.22.1 ISO 439:1982, Gravimetric method

(1) Scope

Determination of total silicon contents between 0,10 % (m/m) and 8,0 % (m/m) in steel and cast iron.

(2) Principle

Dissolution of a test portion in hydrochloric and nitric acids.

Conversion of silica into hydrated silica by fuming with perchloric acid.

Filtration of the hydrated silica, ignition of the impure silica and then weighing.

Treatment of the ignited residue with hydrofluoric and sulfuric acids, followed by ignition and weighing.

3.22.2 ISO 4829-1: 1986, Reduced molybdosilicate spectrophotometric method - Part 1: Silicon contents between 0,05 and 1,0 %

(1) Scope

Determination of total silicon contents between 0,05 % (m/m) and 1,0 % (m/m) in steel and iron soluble in one or the other of the alternative acid mixtures.

(2) Principle

Dissolution of a test portion in an acid mixture appropriate to the alloy composition.

Fusion of the acid-insoluble residue with sodium peroxide.

Formation of the oxidized molybdosilicate (yellow) in weak acid solution.

Selective reduction of the molybdosilicate complex to reduced blue complex with ascorbic acid, after increasing the sulfuric acid concentration and adding oxalic acid to prevent the interference of phosphorus, arsenic and vanadium.

Spectrophotometric measurement of the reduced blue complex at a wavelength of about 810 nm.

(3) Precision: see informative annex A.

Figure A.22.1; Precision for determination of silicon content by ISO 4829-1

3.22.3 ISO 4829-2: 1988, Reduced molybdosilicate spectrophotometric method - Part 2: Silicon contents between 0,01 and 0,05 %

(1) Scope

Determination of total silicon contents between 0,01 % (m/m) and 0,05 % (m/m) in steel and iron.

(2) Principle

Dissolution of a test portion in a hydrochloric acid/nitric acid mixture.

Fusion of the acid-insoluble residue with sodium peroxide.

Formation of the oxidized molybdosilicate (yellow) complex in weak acid solution.

Selective reduction of the molybdosilicate complex to a blue complex with ascorbic acid, after increasing the sulfuric acid concentration and adding oxalic acid to prevent the interference of phosphorus, arsenic and vanadium.

Spectrophotometric measurement of the reduced blue complex at a wavelength of about 810 nm.

(3) Precision: see table 1.

Table 1

Silicon, % (m/m)			
Content	r	Rw	R
0,008	0,002 0	0,002 8	0,005 1
0,015	0,002 2	0,005 7	0,007 2
0,024	0,004 5	0,005 3	0,005 2
0,043	0,006 0	0,007 0	0,009 8
0,055	0,003 8	0,003 2	0,003 8

3.23 Sn Tin

to be prepared.

3.24 Ta Tantalum

to be prepared.

3.25 Ti Titanium

3.25.1 ISO/DIS 10280: Diantipyrylmethane spectrophotometric method

(1) Scope

Determination of titanium contents between 0,002 % (m/m) and 0,80 % (m/m) in steel and iron.

(2) Principle

Dissolution of a test portion with hydrochloric, nitric and sulfuric acid.

Fusion of the residue with potassium hydrogen sulfate.

Formation of a yellow complex with 4,4'-diantipyrylmethane.

Spectrophotometric measurement of the coloured complex at a wavelength of about 385 nm.

(3) Precision: see informative annex A.

Figure A.25.1; Precision for determination of titanium content by ISO/DIS 10280

3.26 V Vanadium

3.26.1 ISO 4942: 1988, N-BPHA spectrophotometric method

(1) Scope

Determination of vanadium contents between 0,005 % (m/m) and 0,50 % (m/m) in steel and iron.

(2) Principle

Dissolution of a test portion with appropriate acids.

Addition of orthophosphoric acid to an aliquot of the solution thus obtained to prevent the interference of iron, and addition of potassium permanganate to oxidize vanadium to the pentavalent state.

Selective reduction of excess permanganate by sodium nitrite in the presence of urea and treatment with N-BPHA and hydrochloric acid to form a complex, followed by extraction of the complex into trichloromethane.

Spectrophotometric measurement of the absorbance at approximately 535 nm.

(3) Precision: see informative annex A.

Figure A.26.1; Precision for determination of vanadium content by ISO 4942

3.26.2 ISO 4947: 1986, Potentiometric titration method

(1) Scope

Determination of vanadium contents between 0,04 % (m/m) and 2 % (m/m) in steel and cast iron.

(2) Principle

Dissolution of a test portion with appropriate acids.

Addition of hydrofluoric acid to keep tungsten in solution.

Oxidation of chromium and vanadium by potassium peroxydisulfate. Partial oxidation of chromium.

While checking the potential of the solution,

- reduction of chromium(VI) and vanadium(V) by ammonium iron(II) sulfate;
- oxidation of vanadium by slight excess of potassium permanganate; reduction of the excess permanganate by sodium nitrite, and reduction of the excess sodium nitrite by sulfamic acid.

Potentiometric titration of vanadium by ammonium iron(II) sulfate standard solution.

(3) Precision: see informative annex A.

Figure A.26.2; Precision for determination of vanadium content by ISO 4947

3.26.3 ISO 9647: 1989, Flame atomic absorption spectrometric method

(1) Scope

Determination of vanadium contents between 0,005 % (m/m) and 1,0 % (m/m) in steel and iron, provided that tungsten content in the test portion is not higher than 10 mg and/or titanium is not higher than 5 mg.

(2) Principle

Dissolution of a test portion in hydrochloric, nitric and perchloric acids.

Addition of aluminium solution as spectrochemical buffer.

Spraying of the solution into a dinitrogen monoxide-acetylene flame.

Spectrometric measurement of the atomic absorption of the 318,4 nm spectral line emitted by a vanadium hollow cathode lamp.

(3) Precision: see informative annex A.

Figure A.26.3; Precision for determination of vanadium content by ISO 9647

3.27 **W** Tungsten

to be prepared.

3.28 **Zr** Zirconium

to be prepared.

Informative annex A

Graphical representation of precision data

A.1.1 **Aluminium** (acid-soluble) - Flame atomic absorption spectrometric method - ISO 9658: 1990

A.1.2 **Aluminium** (total) - Flame atomic absorption spectrometric method - ISO 9658: 1990

A.3.1 **Boron** - Curcumin spectrophotometric method - ISO 10153: 1991

A.5.1 **Carbon** (total) - Infrared absorption method after combustion in an induction furnace - ISO 9556: 1989

A.6.1 **Calcium** (acid-soluble) - Flame atomic absorption spectrometric method - ISO/DIS 10697-1

A.9.1 **Chromium** - Potentiometric titration method - ISO 4937: 1986

A.9.2 **Chromium** - Visual titration method - ISO 4937: 1986

A.9.3 **Chromium** - Flame atomic absorption spectrometric method - ISO/DIS 10138

A.10.1 **Copper** - Flame atomic absorption spectrometric method - ISO 4943: 1985

A.10.2 **Copper** - 2,2'-Diquinolyl spectrophotometric method - ISO 4946: 1984

A.12.1 **Manganese** - Flame atomic absorption spectrometric method - ISO/TR 10281: 1990

A.15.1 **Niobium** - PAR spectrophotometric method - ISO 9441: 1988

A.16.1 **Nickel** - Gravimetric method - ISO 4938: 1988

A.16.2 **Nickel** - Titrimetric method - ISO 4938: 1988

A.16.3 **Nickel** - Dimethylglyoxime spectrophotometric method - ISO 4939: 1984

A.16.4 **Nickel** - Flame atomic absorption spectrometric method - ISO 4940: 1985

A.20.1 **Sulfur** - Infrared absorption method after combustion in an induction furnace - ISO 4935: 1989

A.22.1 **Silicon** (total) - Reduced molybdosilicate spectrophotometric method - ISO 4829-1: 1986

A.25.1 **Titanium** - Diantipyrylmethane spectrophotometric method - ISO/DIS 10280

A.26.1 Vanadium - N-BPHA spectrophotometric method - ISO 4942: 1988

A.26.2 Vanadium - Potentiometric titration method - ISO 4947: 1986

A.26.3 Vanadium - Flame atomic absorption spectrometric method - ISO 9647: 1989

NOTE - The types of precision (r, Rw and R) and the conditions under which the tests were carried out are listed in informative annex B.

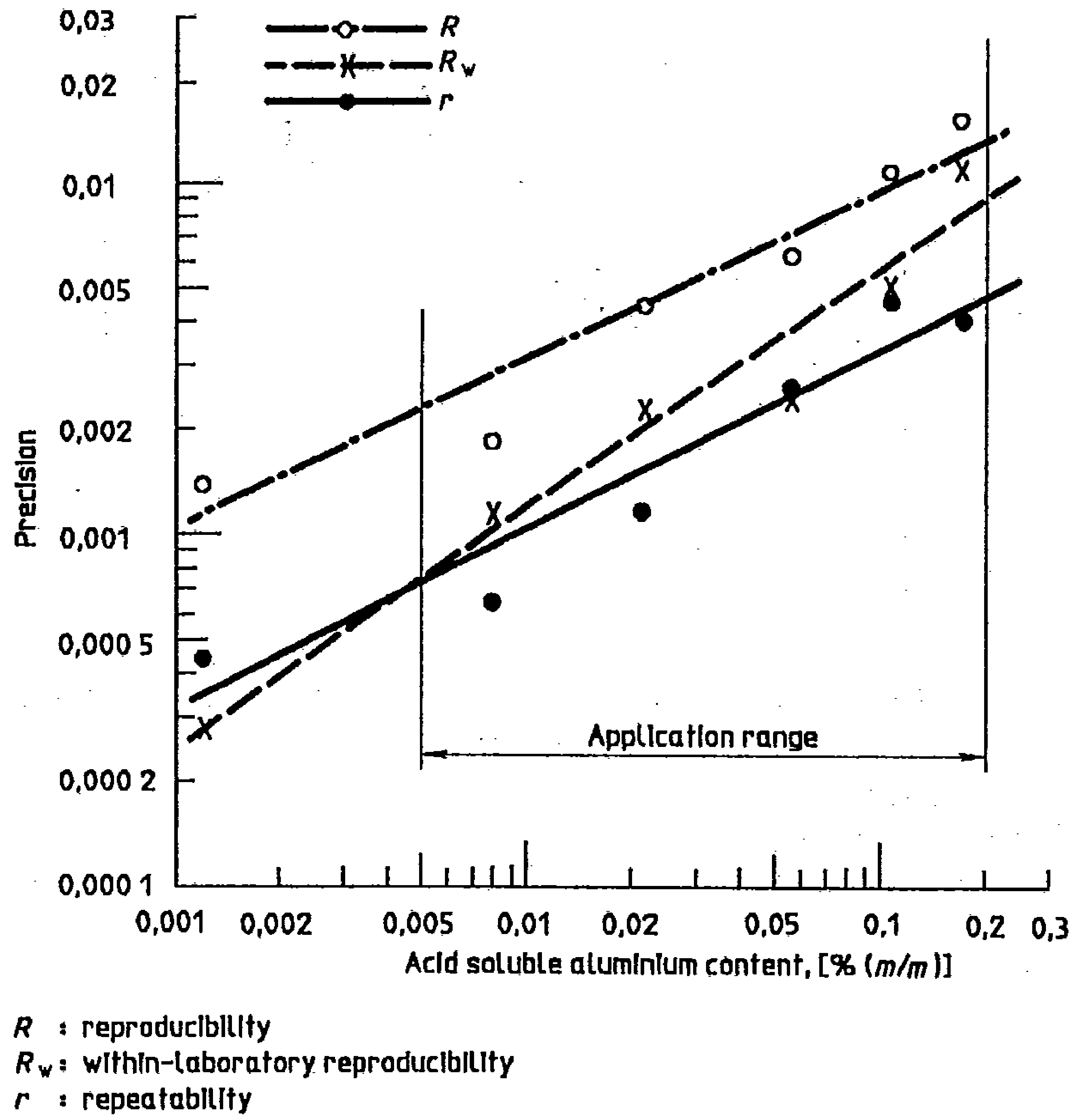


Figure A.1.1 — Precision for determination of acid-soluble aluminium content — Flame atomic absorption spectrometric method

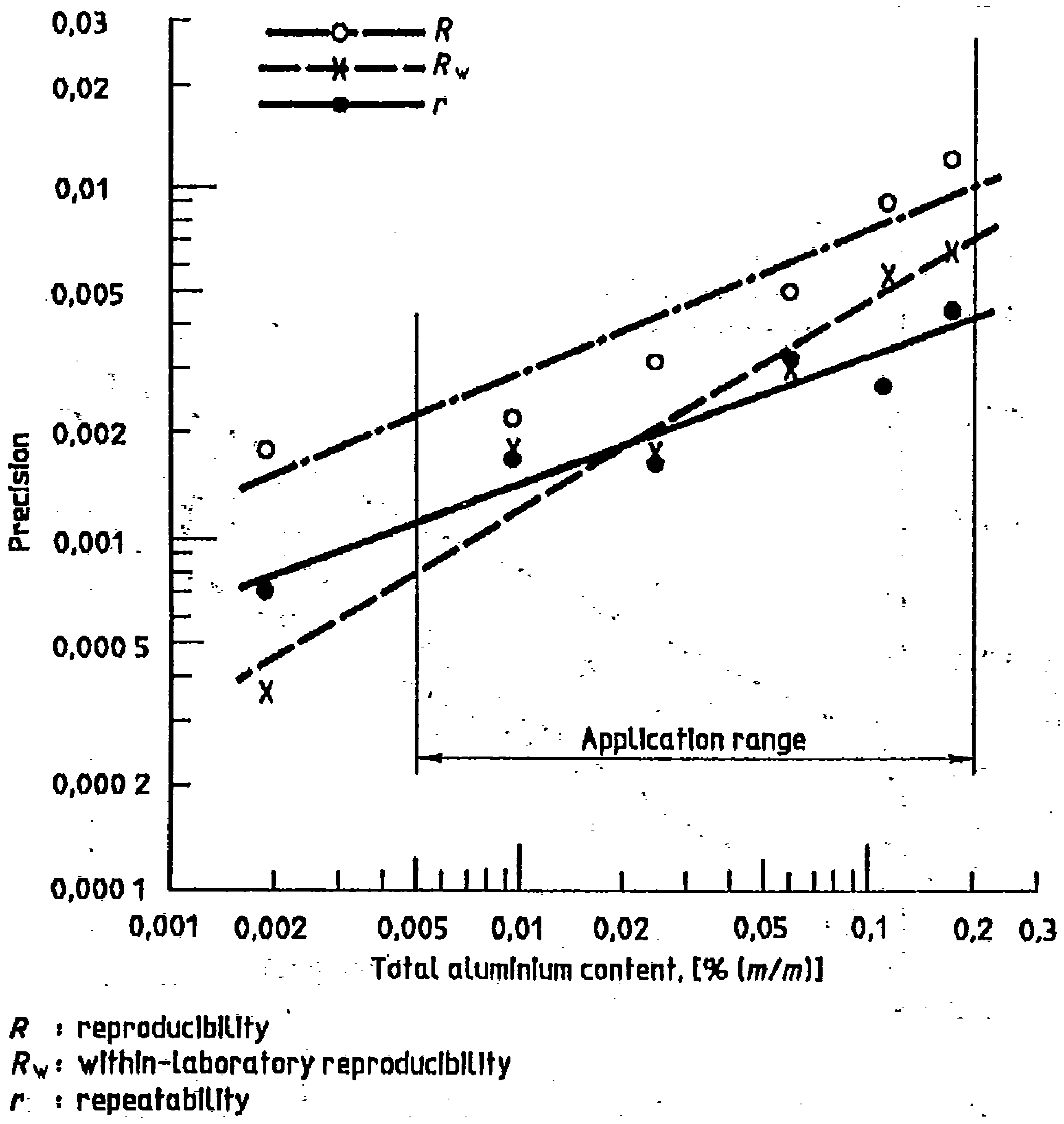
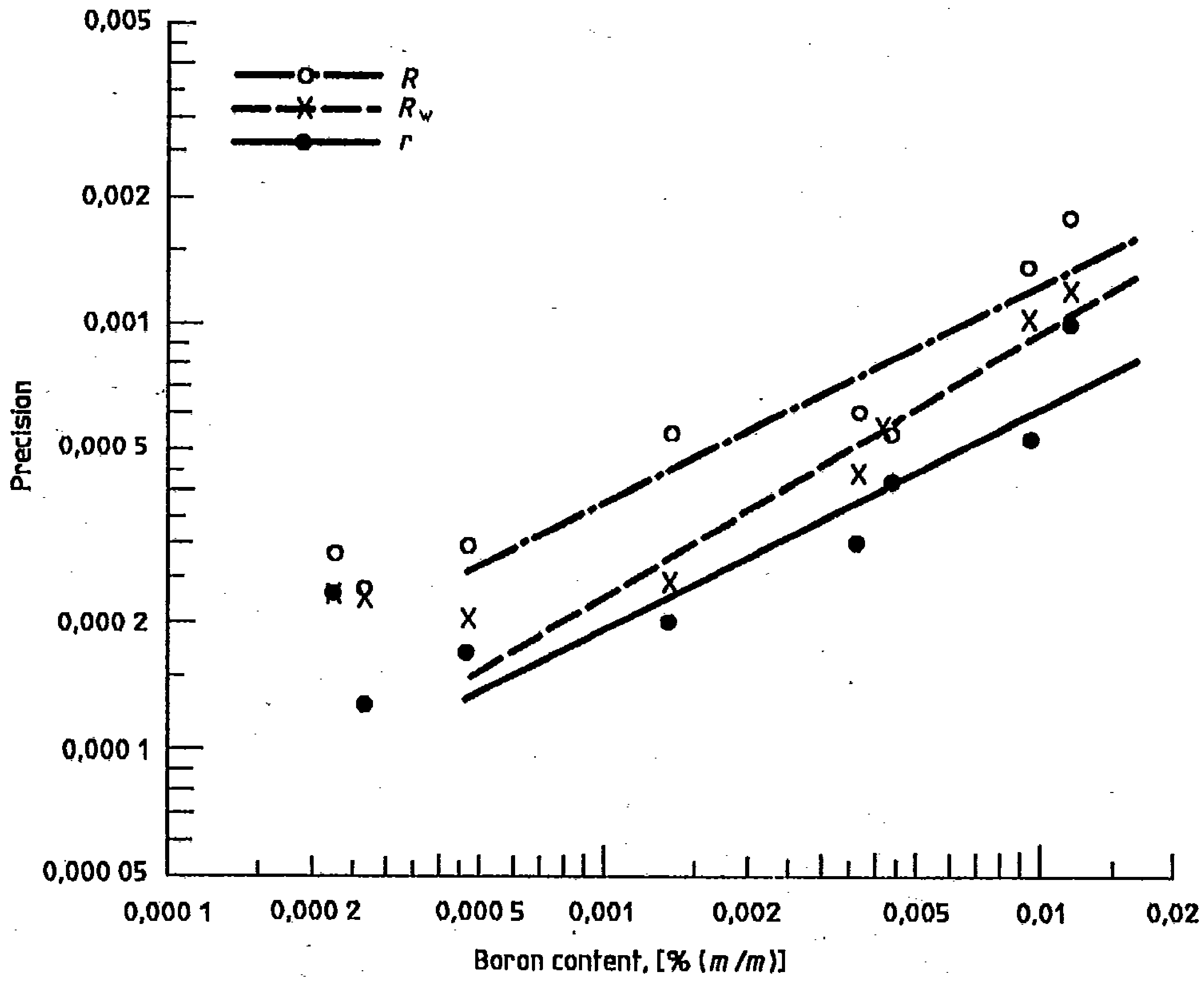
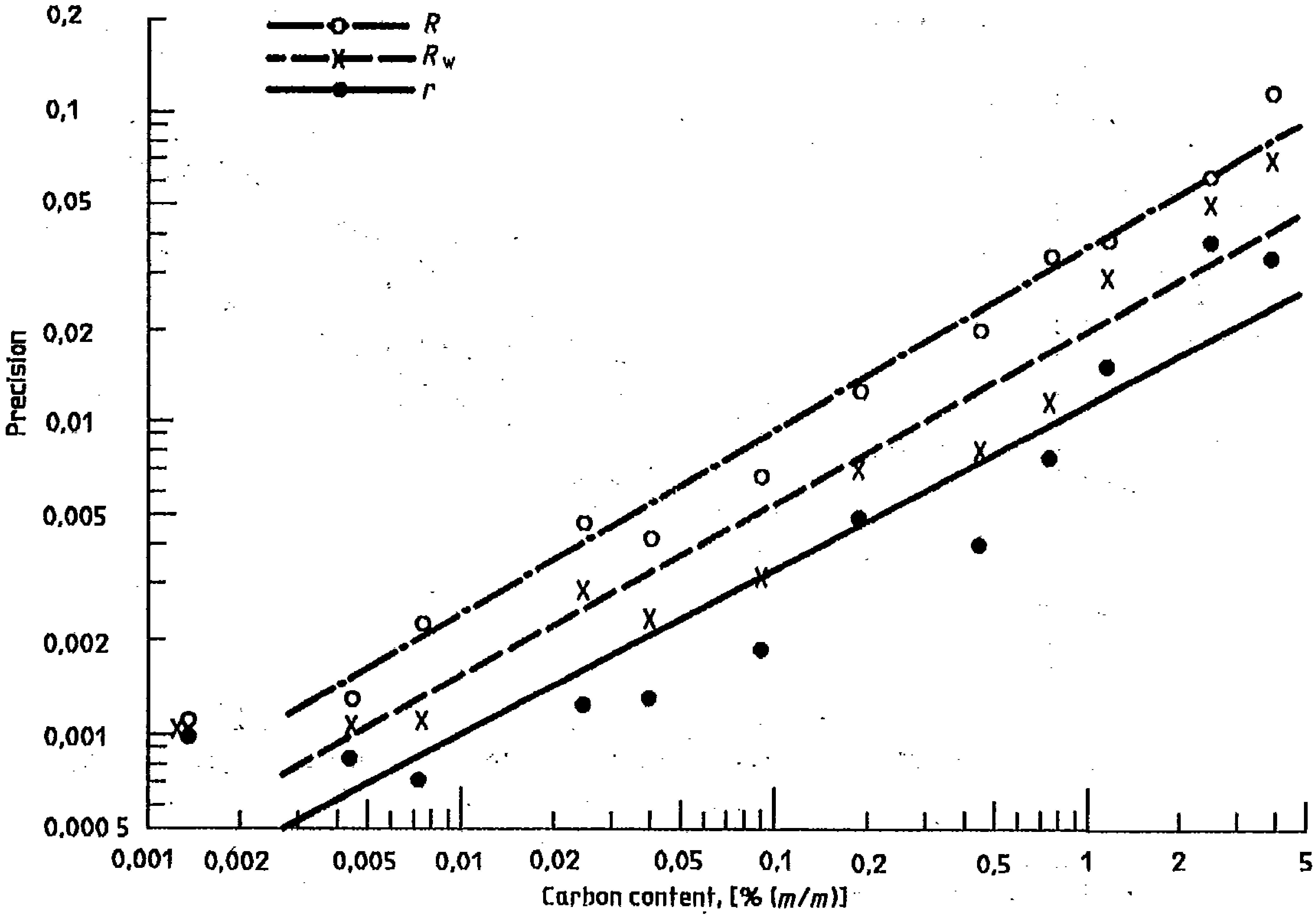


Figure A.1.2 — Precision for determination of total aluminium content — Flame atomic absorption spectrometric method



R : reproducibility
R_w: within-laboratory reproducibility
r : repeatability

Figure A.3.1 — Precision for determination of boron content — Curcumin spectrophotometric method

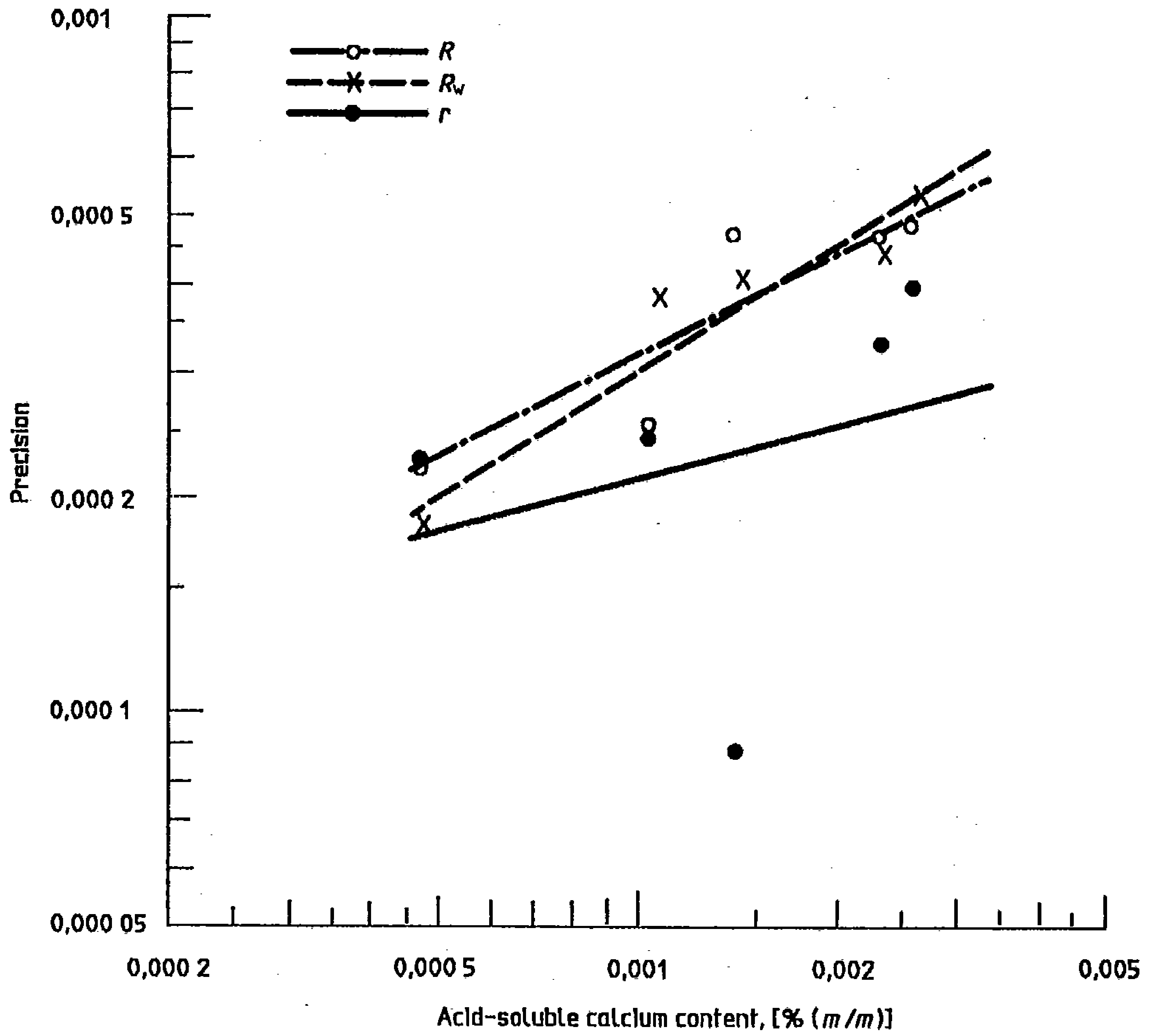


R : reproducibility

R_w : within-laboratory reproducibility

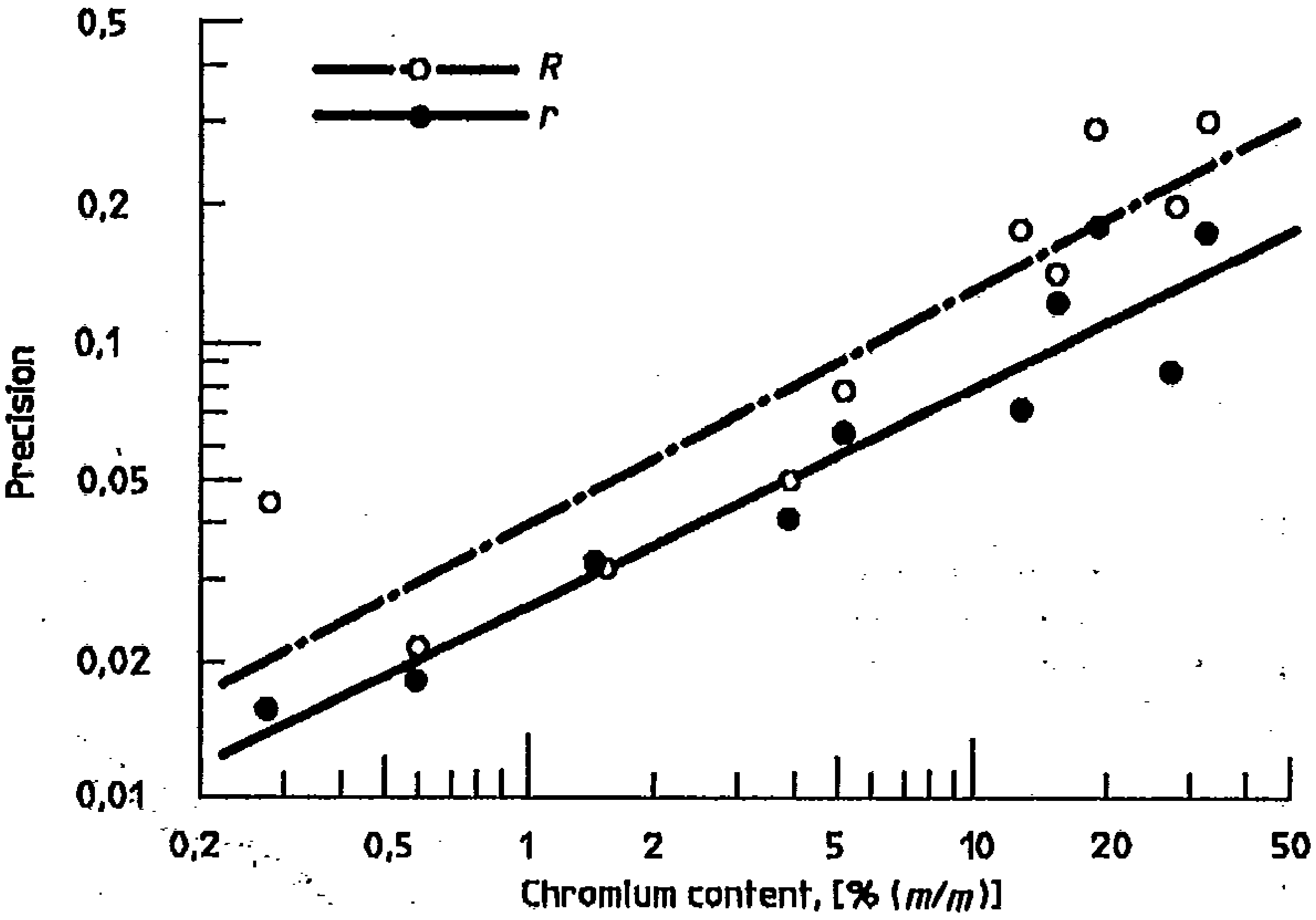
r : repeatability

Figure A.5.1 — Precision for determination of total carbon content — Infrared absorption method after combustion in an induction furnace



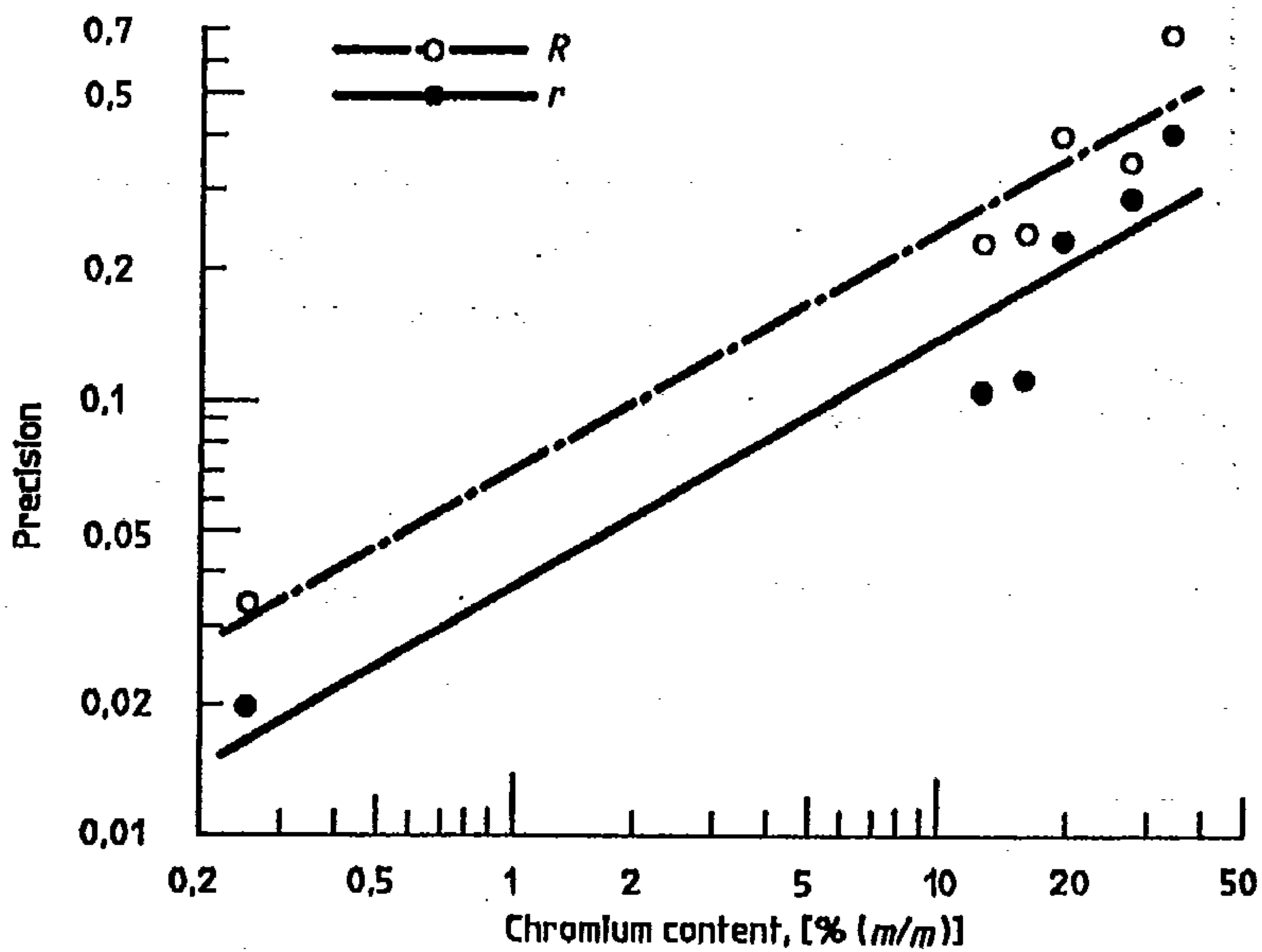
R : reproducibility
R_w : within-laboratory reproducibility
r : repeatability

Figure A.6 1 — Precision for determination of acid-soluble calcium content — Flame atomic absorption spectrometric method



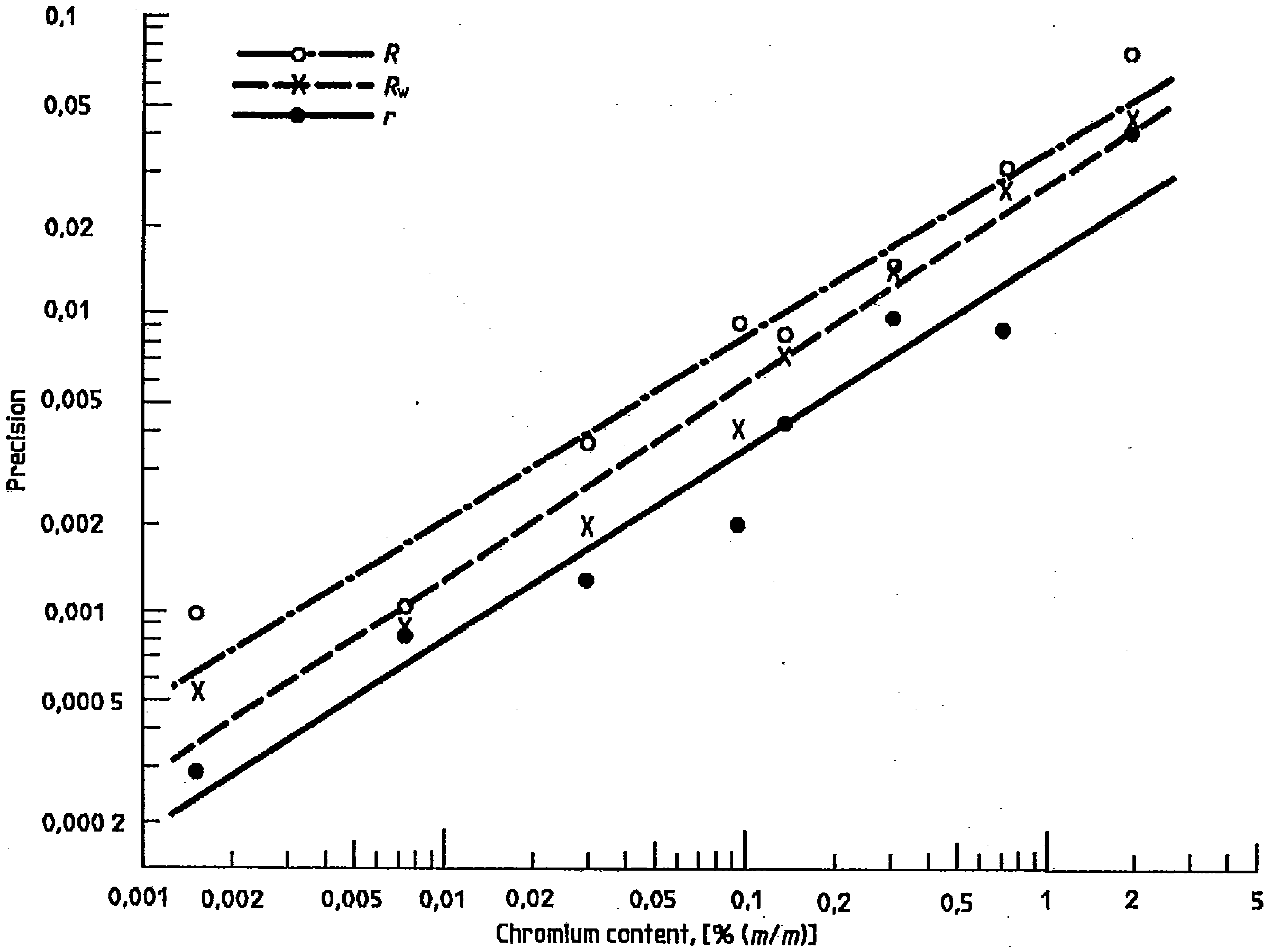
R : reproducibility
r : repeatability

Figure A.9.1 — Precision for determination of chromium content — Potentiometric titration method



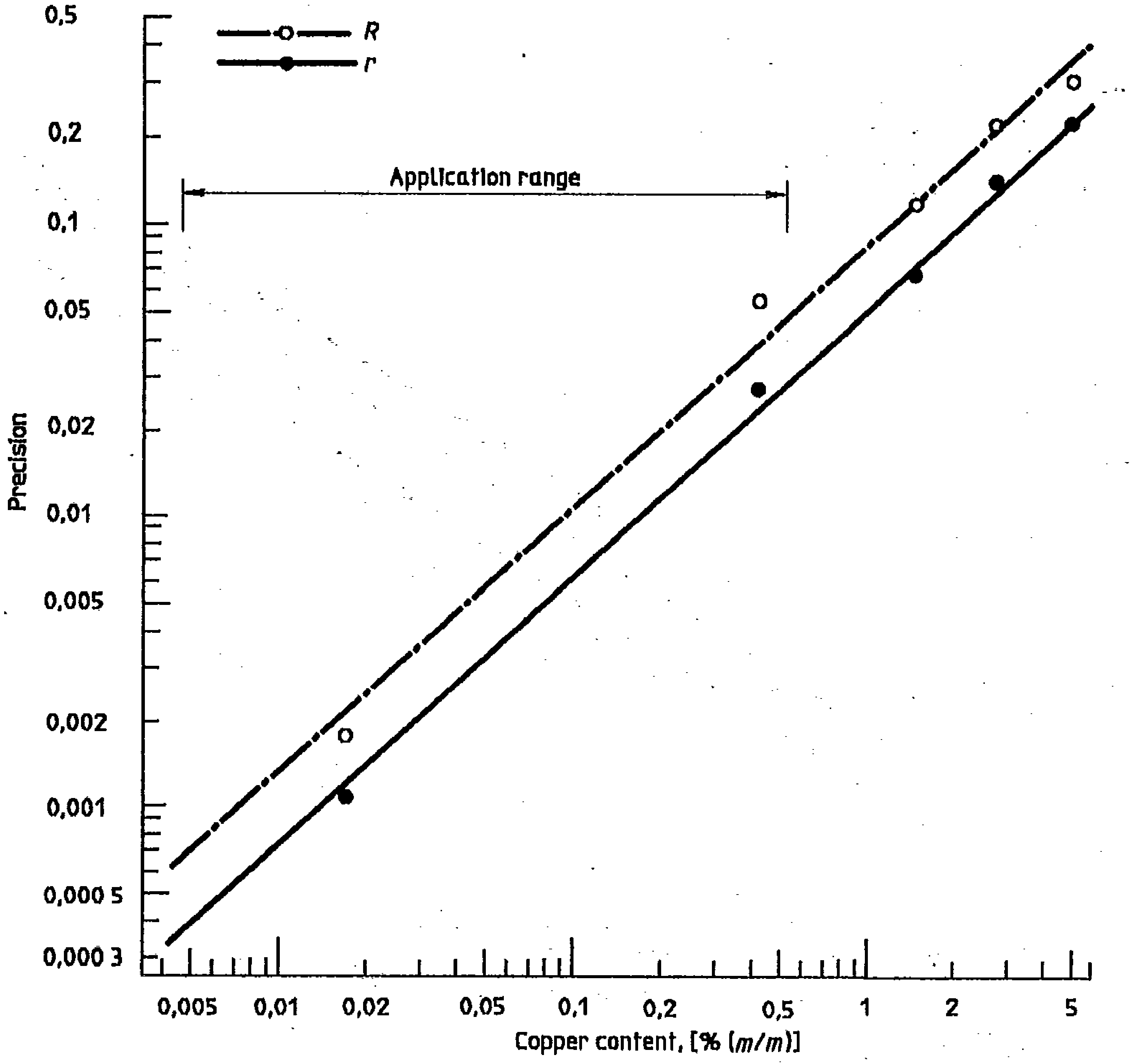
R : reproducibility
r : repeatability

Figure A.9.2 — Precision for determination of chromium content — Visual titration method



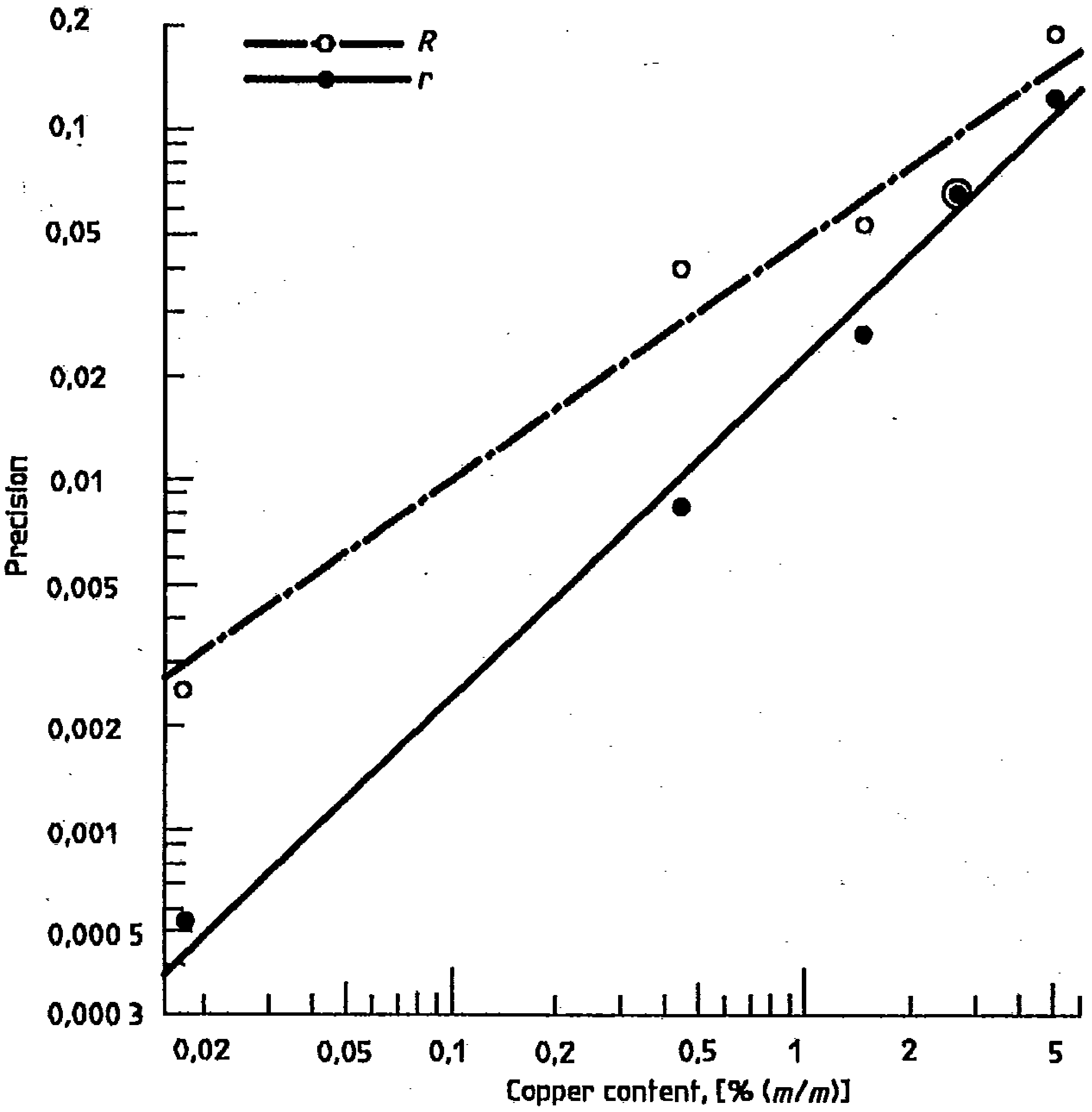
R : reproducibility
R_w: within-laboratory reproducibility
r : repeatability

Figure A.9.3 — Precision for determination of chromium content — Flame atomic absorption spectrometric method



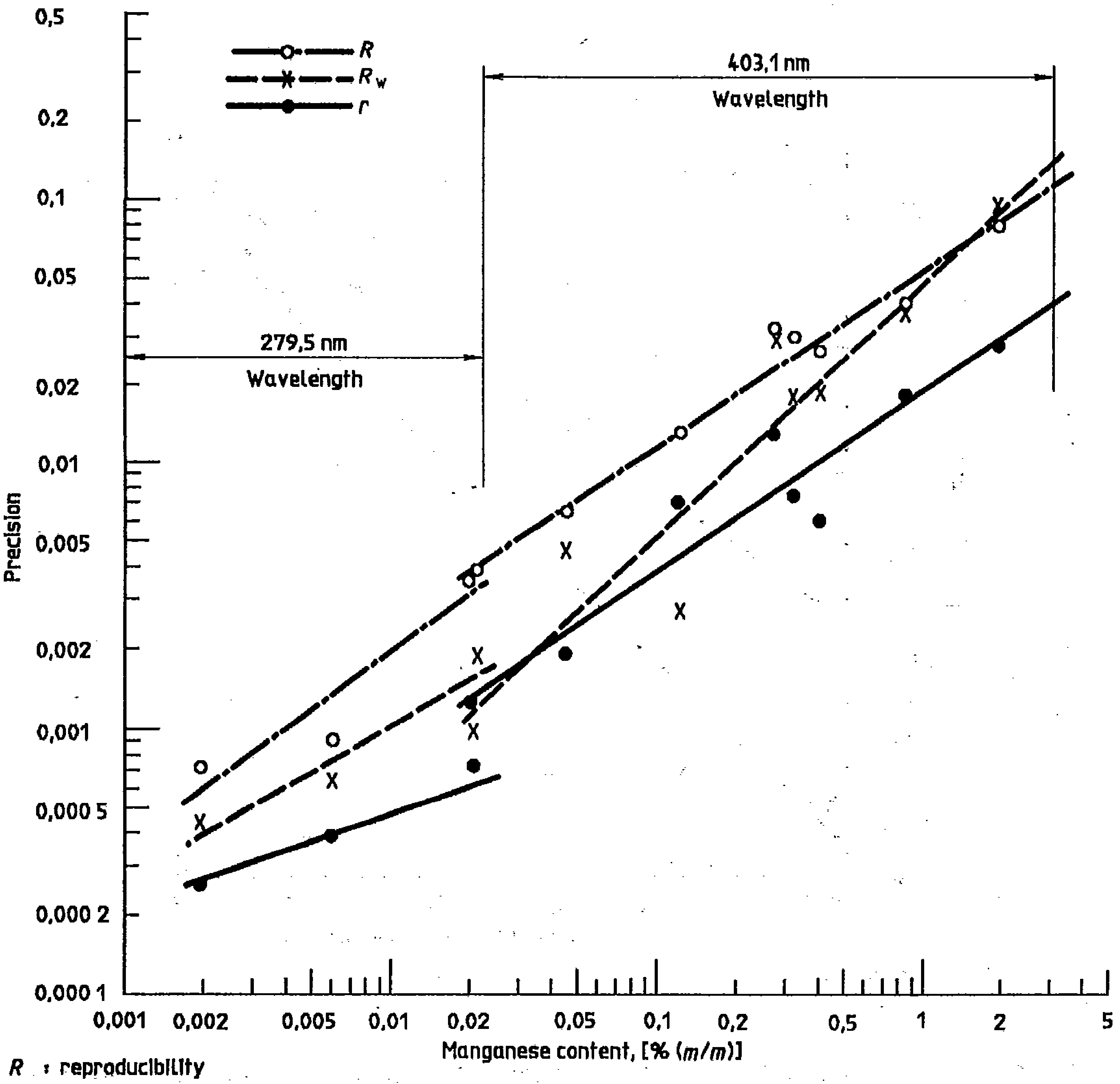
R : reproducibility
r : repeatability

Figure A.10.1 — Precision for determination of copper content — Flame atomic absorption spectrometric method



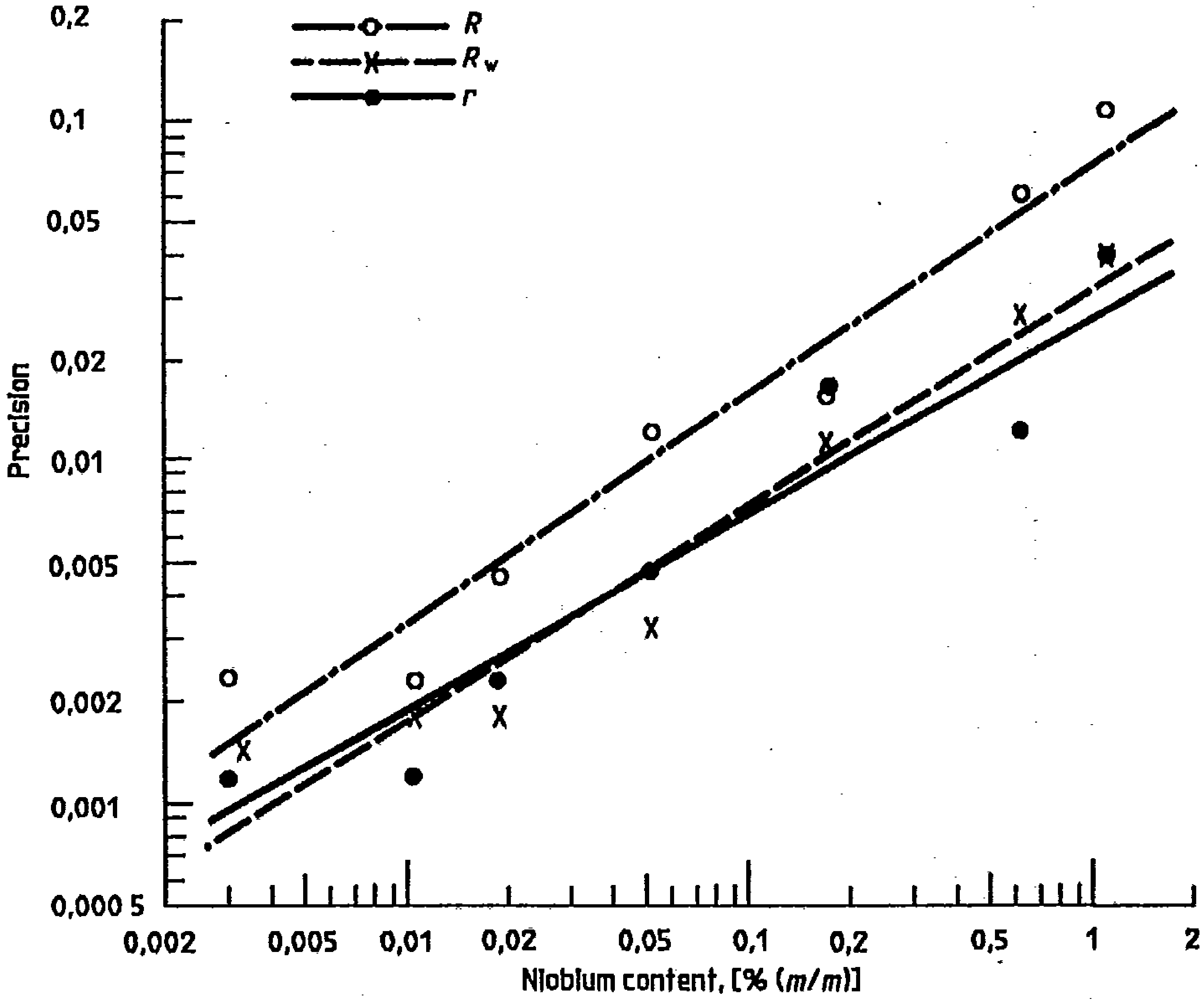
R : reproducibility
r : repeatability

Figure A.10.2 — Precision for determination of copper content — 2,2′-diquinolyl spectrophotometric method



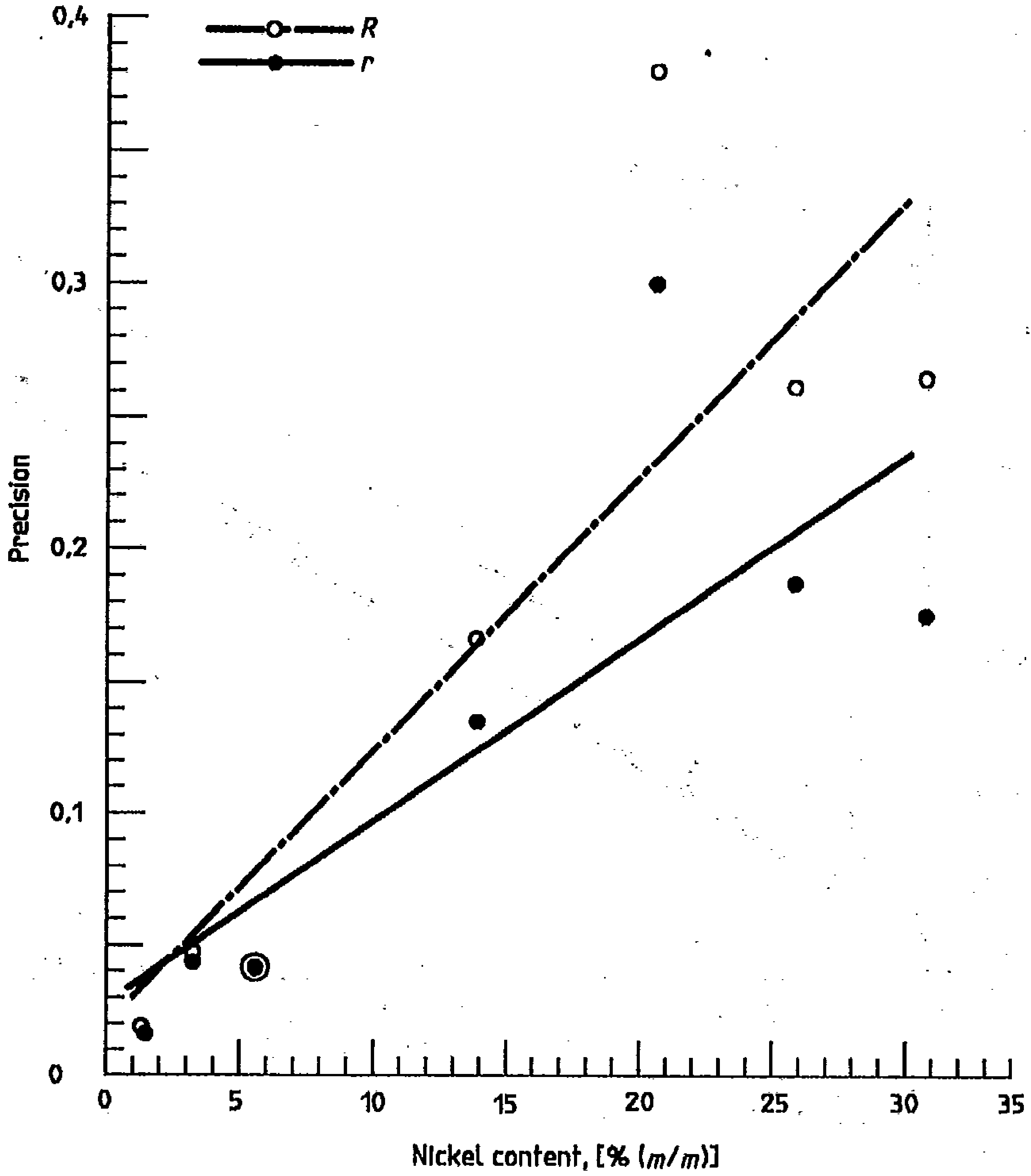
R : reproducibility
R_w : within-laboratory reproducibility
r : repeatability

Figure A.12.1 — Precision for determination of manganese content — Flame atomic absorption spectrometric method (TR 10281)



R : reproducibility
R_w : within-laboratory reproducibility
r : repeatability

Figure A.15.1 — Precision for determination of niobium content — PAR spectrophotometric method



R : reproducibility
r : repeatability

Figure A.16.1 — Precision for determination of nickel content — Gravimetric method

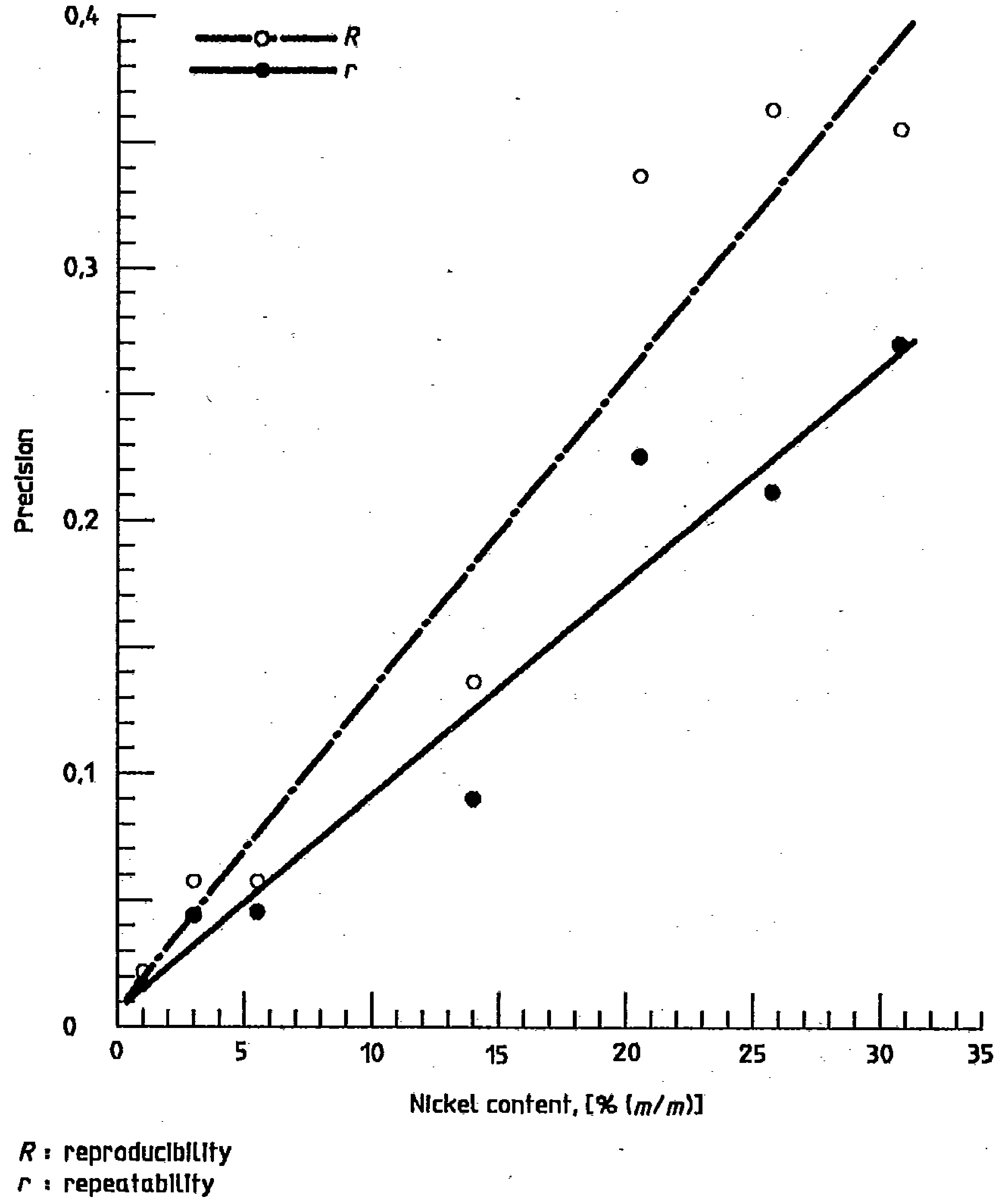


Figure A.16.2 — Precision for determination of nickel content — Titrimetric method

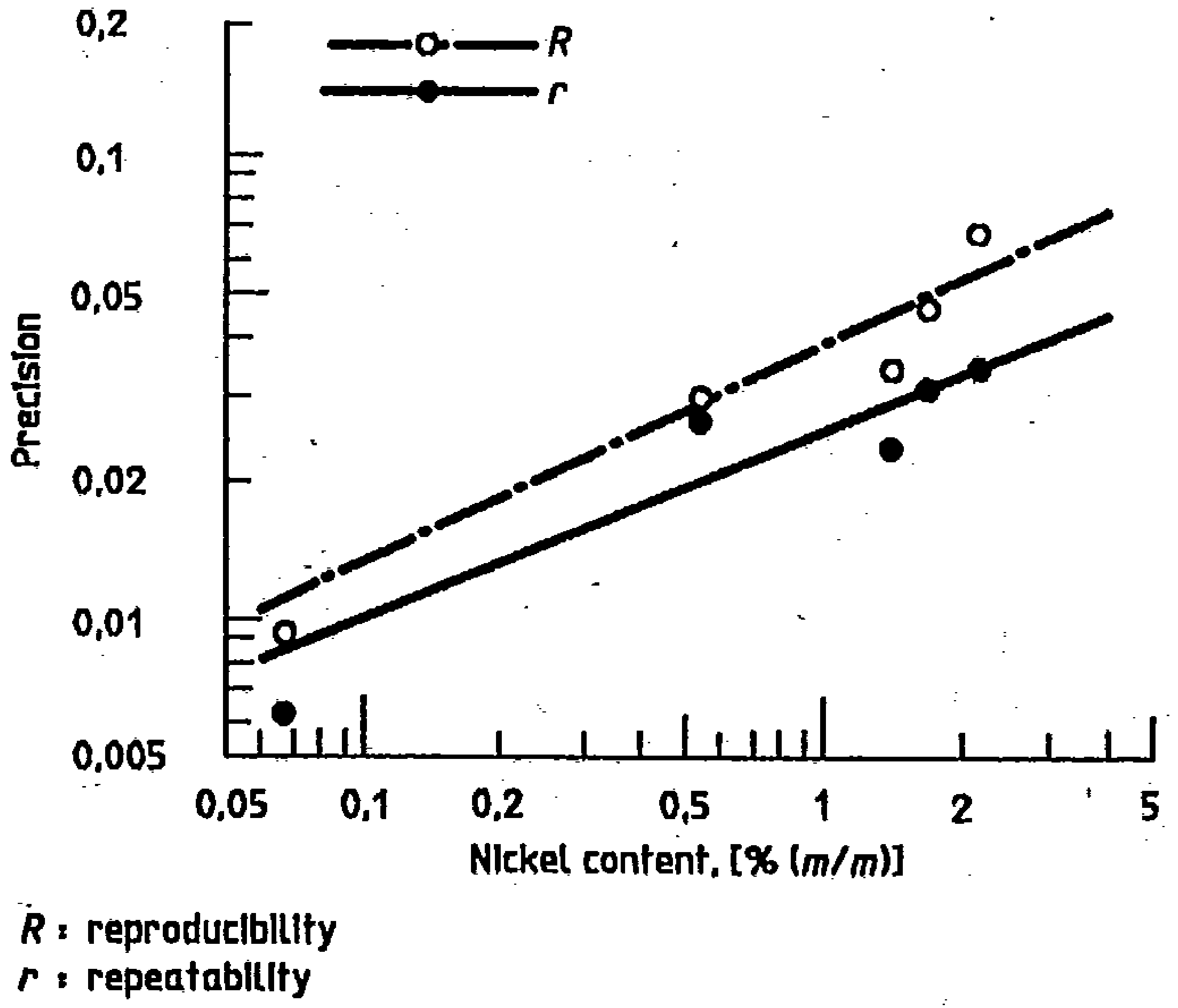


Figure A.16.3 — Precision for determination of nickel content — Dimethylglyoxime spectrophotometric method

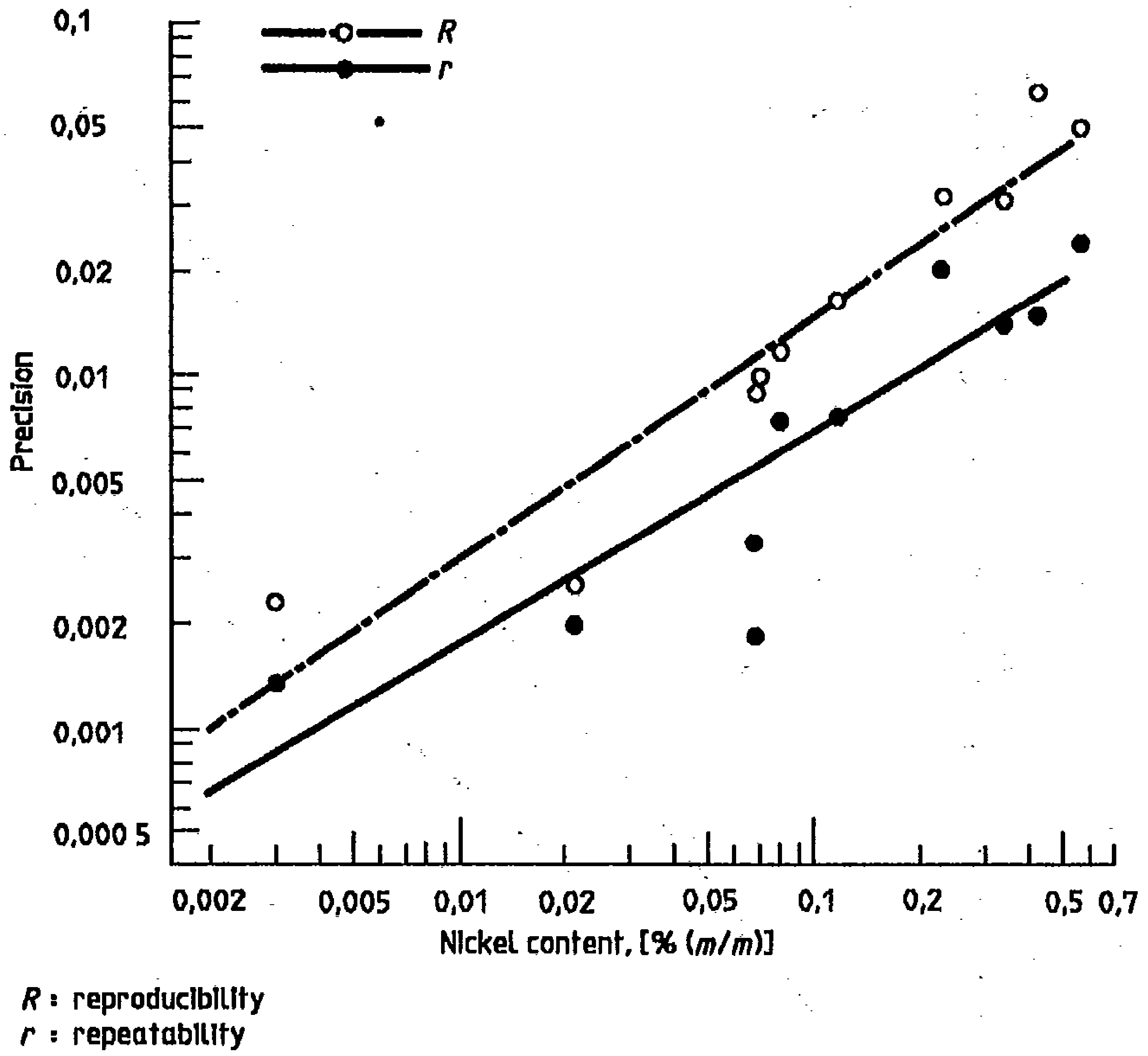


Figure A.16.4 — Precision for determination of nickel content — Flame atomic absorption spectrometric method

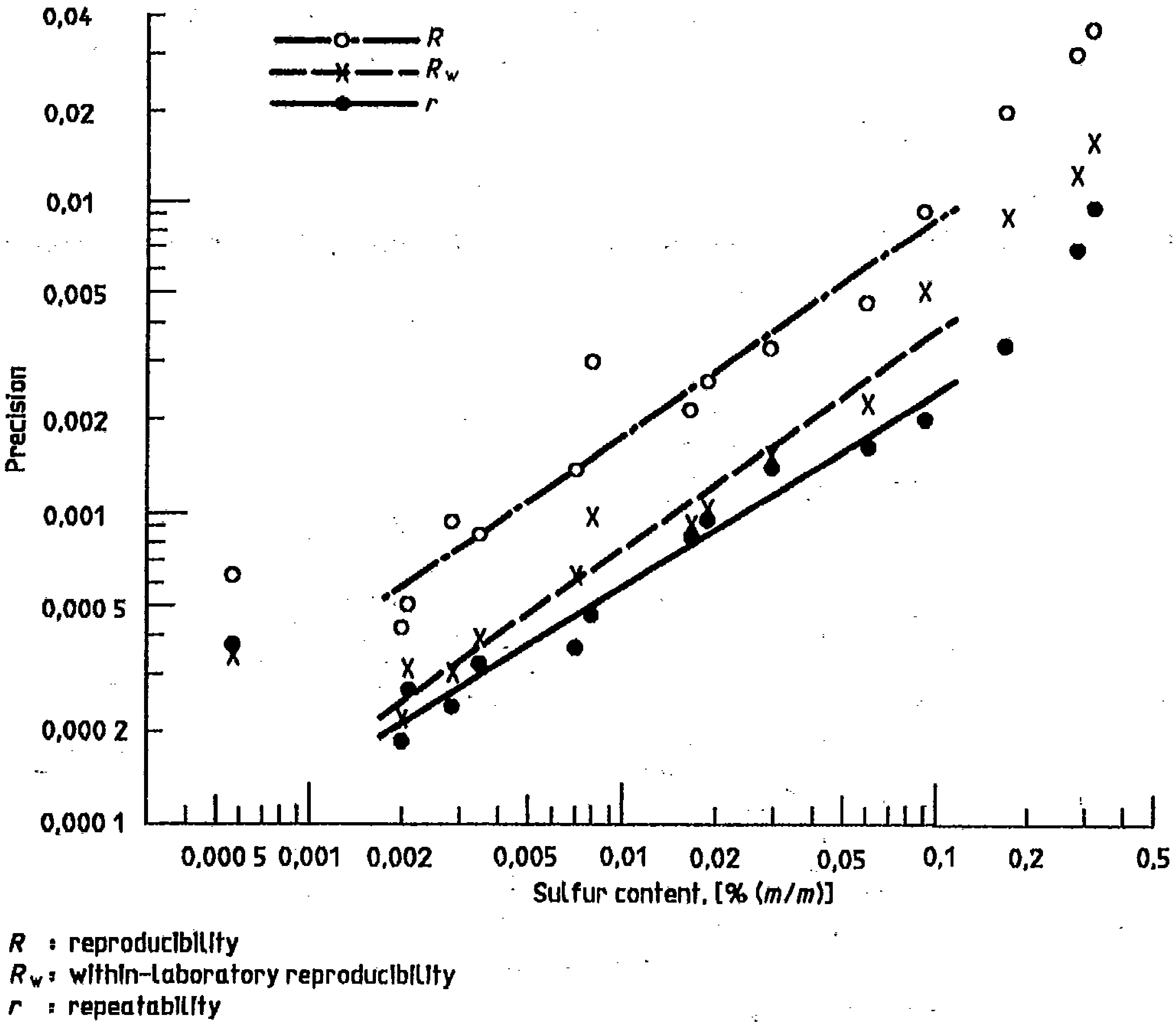


Figure A.20.1 — Precision for determination of sulfur content — Infrared absorption method after combustion in an induction furnace

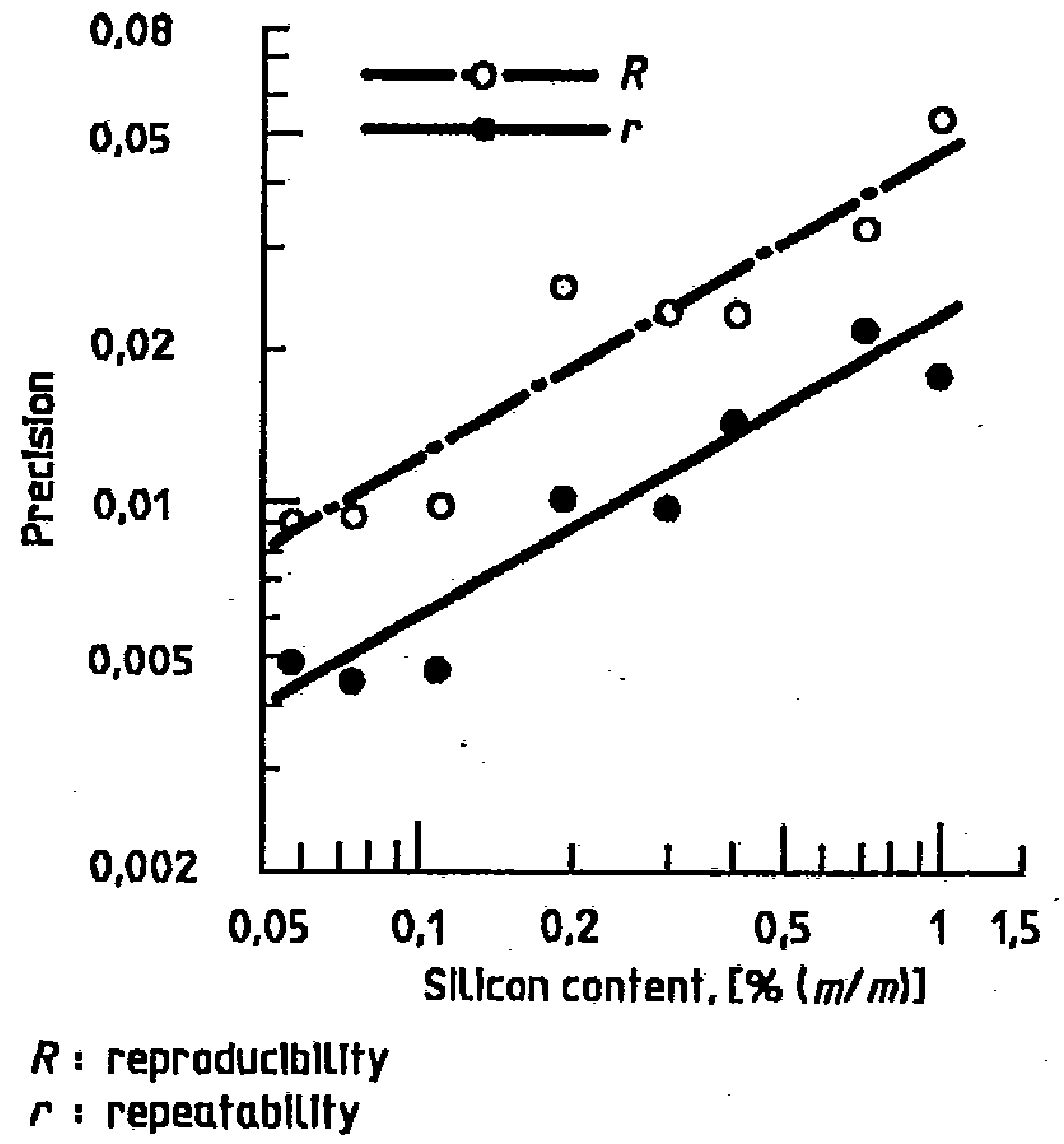


Figure A.22.1 — Precision for determination of silicon content — Reduced molybdosilicate spectrophotometric method — Part 1: Silicon contents between 0,05 and 1,0 %

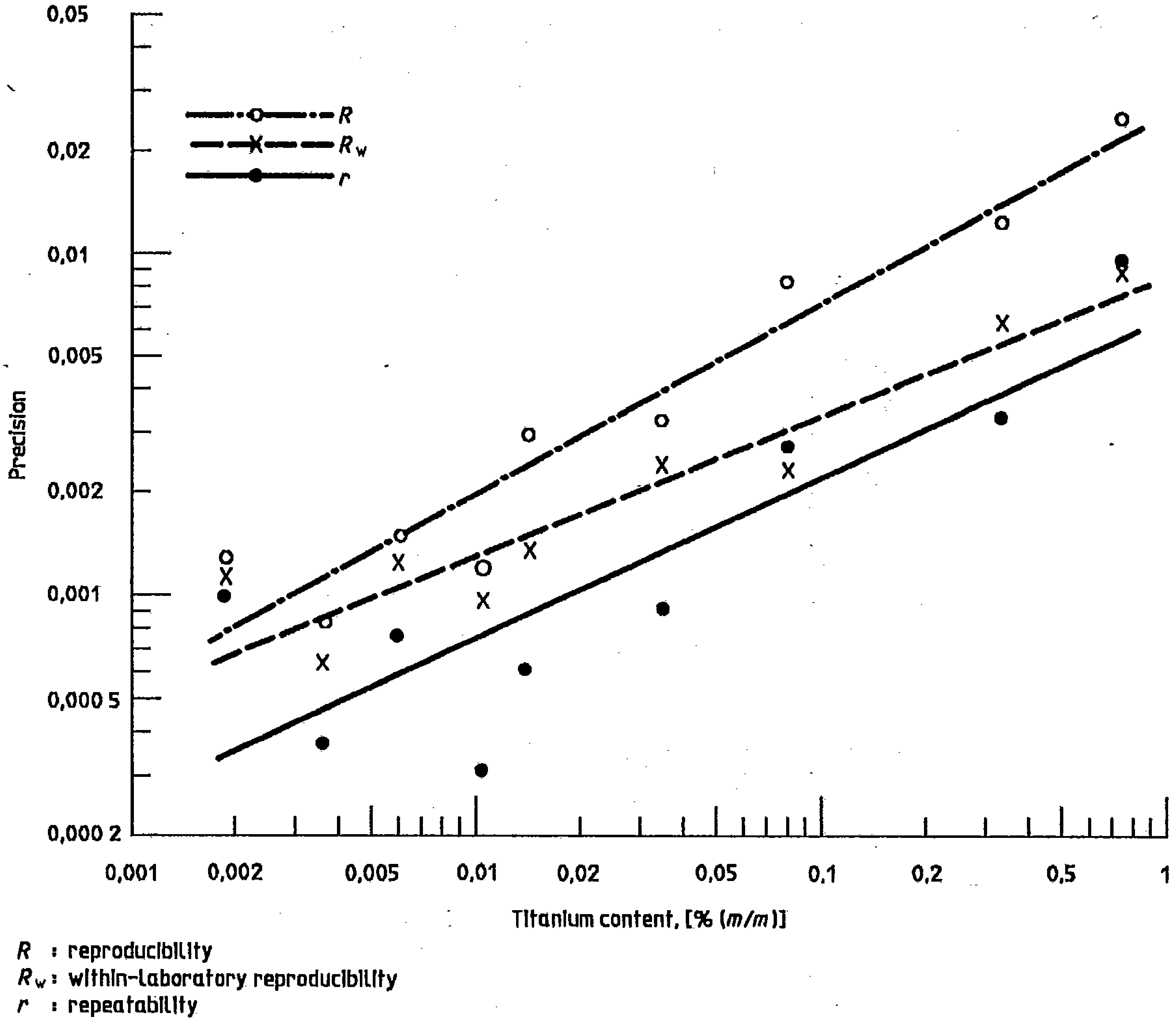
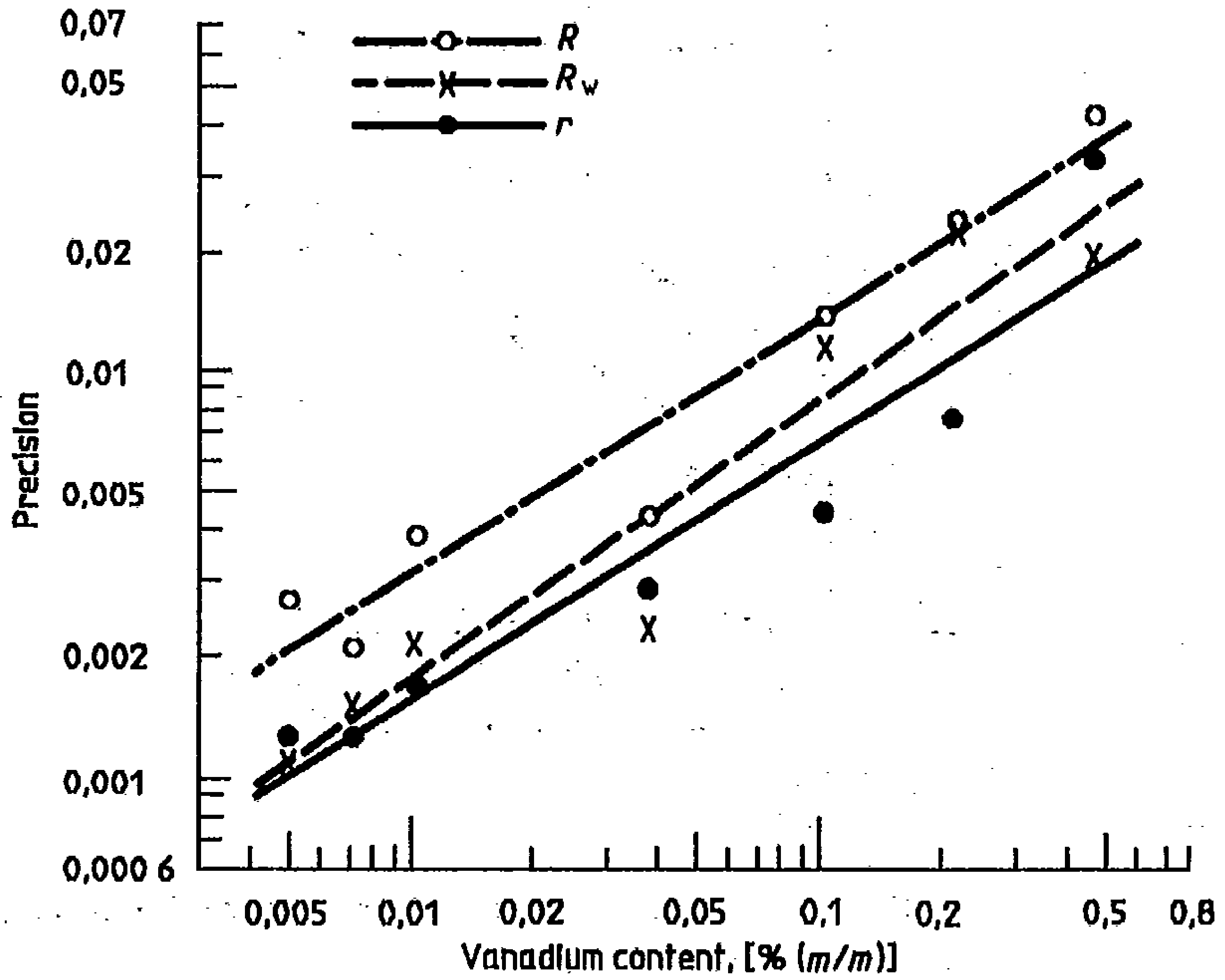
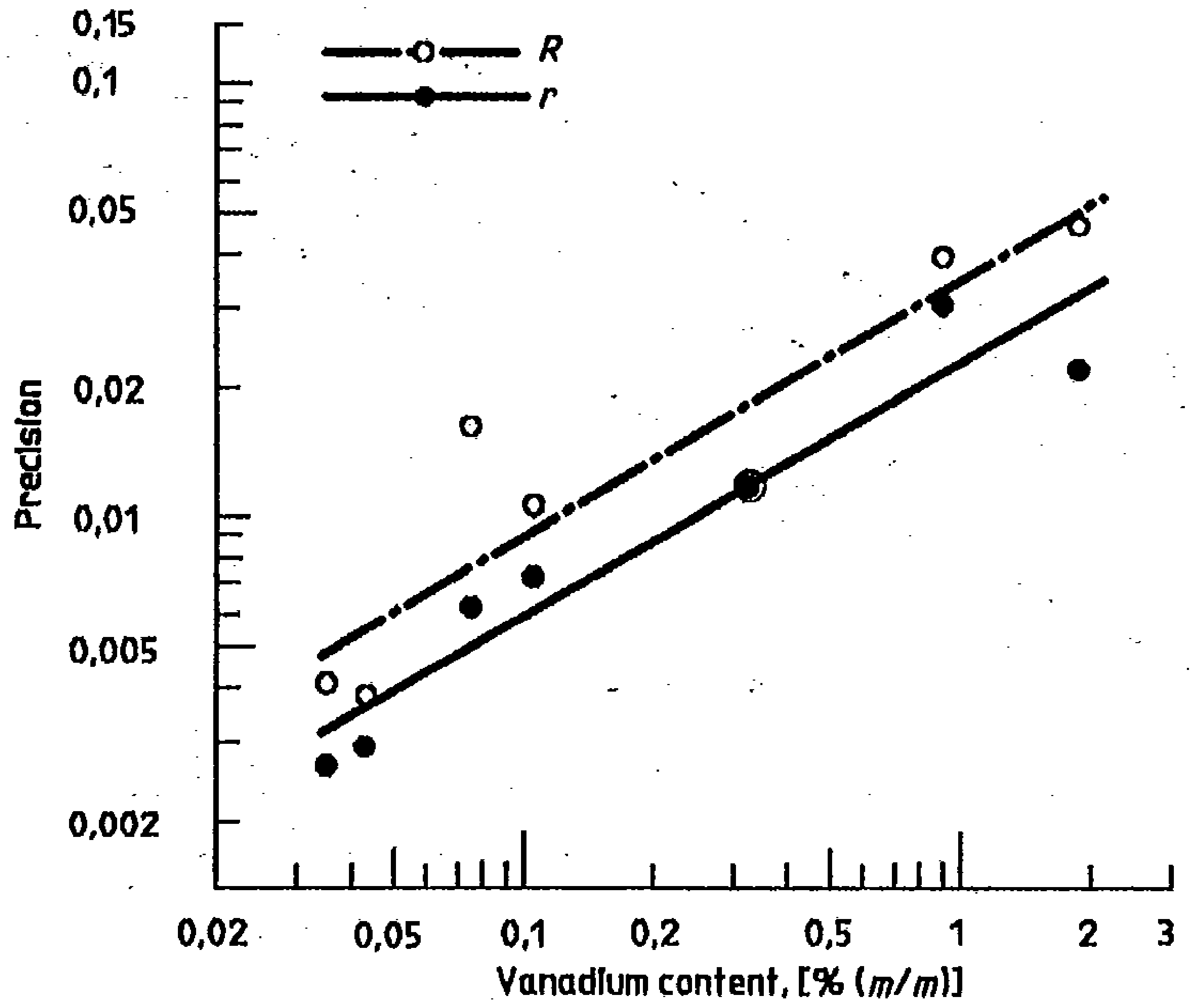


Figure A.25.1 — Precision for determination of titanium content — Diantipyrylmethane spectrophotometric method



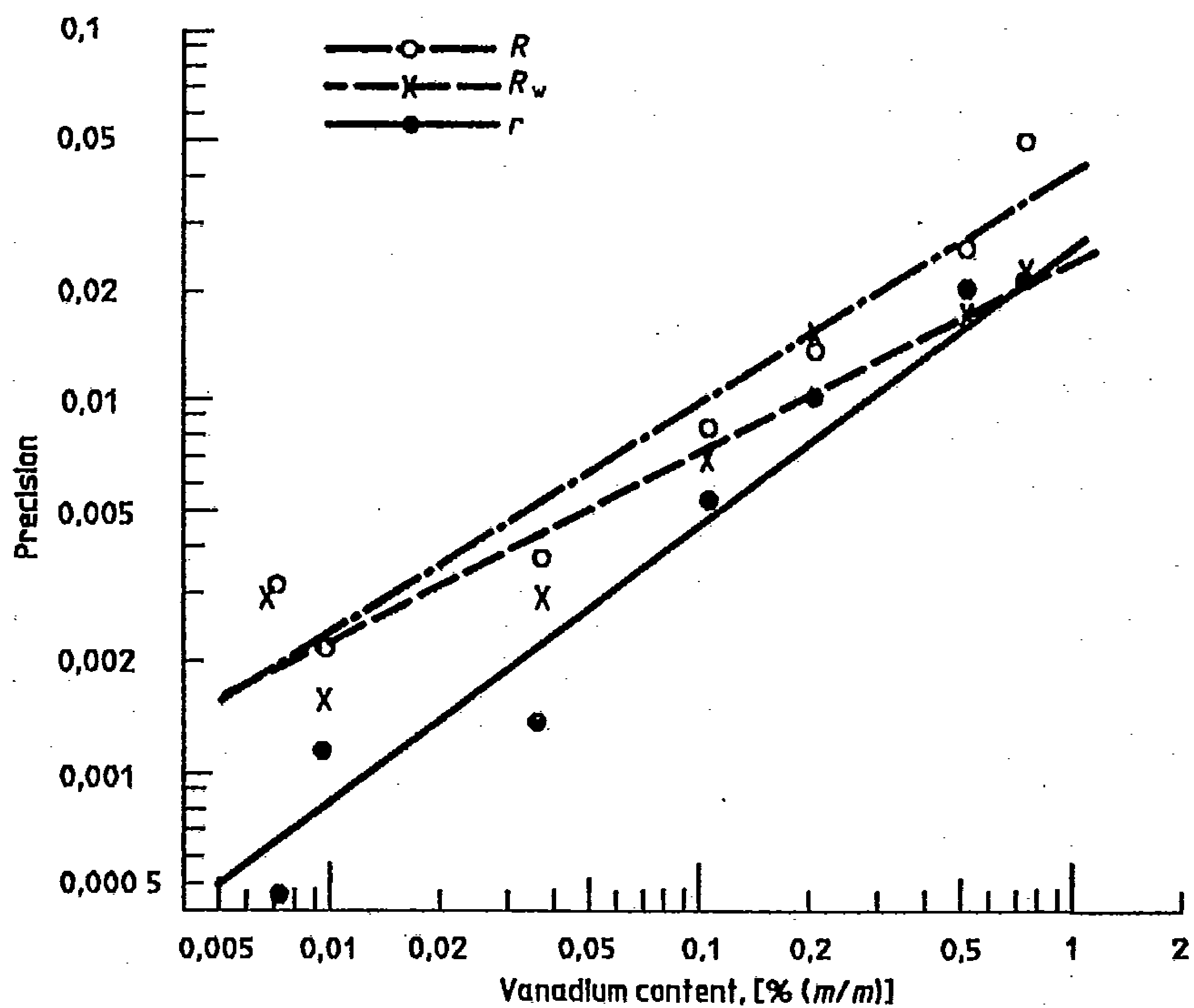
R : reproducibility
R_w : within-laboratory reproducibility
r : repeatability

Figure A.26.1 — Precision for determination of vanadium content — N-BPHA spectrophotometric method



R : reproducibility
r : repeatability

Figure A.26.2 — Precision for determination of vanadium content — Potentiometric titration method



R : reproducibility
R_w: within-laboratory reproducibility
r : repeatability

Figure A.26.3 — Precision for determination of vanadium content — Flame atomic absorption spectrometric method

Informative annex B

Type of precision and its test conditions

Element	Standard(s)	Types of precision1)	Test conditions of determinations	Relevant figure(s)
Al	ISO 9658:1990 - Flame atomic absorption spectrometric method	*r*, *Rw*, *R*	Three determinations at a level2)	A.1.1 A.1.2
B	ISO/DIS 10153 - Curcumin spectrophotometric method	ditto	ditto	A.3.1
C	ISO 9556:1989 - Infrared absorption method after combustion in an induction furnace	ditto	ditto	A.5.1
Ca	(acid-soluble Ca) ISO/DIS 10697-1 - Flame atomic absorption spectrometric method	ditto	ditto	A.6.1
Cr	ISO 4937:1986 - Potentiometric or visual titration method	*r*, *R*	Two independent3) determinations at a level	A.9.1 A.9.2
	ISO/DIS 10138 - Flame atomic absorption spectrometric method	*r*, *Rw*, *R*	Three determinations at a level2)	A.9.3
Cu	ISO 4943:1985 - Flame atomic absorption spectrometric method	*r*, *R*	Four to five determinations at a level One determination at a day	A.10.1
	ISO 4946:1984 - 2,2'-Diquinolyl spectrophotometric method	*r*, *R*	Three to five determinations at a level One determination at a day	A.10.2
Mn	ISO/TR 10281:1990 - Flame atomic absoroption spectrometric method	*r*, *Rw*, *R*	Three determinations at a level2)	A.12.1
Nb	ISO 9441:1988 - PAR spectrophotometric method	ditto	ditto	A.15.1

Element	Standard(s)	Types of precision1)	Test conditions of determinations	Relevant figure(s)
Ni	ISO 4938:1988 - Gravimetric or titrimetric method	r, R	Four independent determinations at a level	A.16.1 A.16.2
	ISO 4939:1984 - Dimethylglyoxime spectrophtometric method	r, R	Six determinations at a level One determination at a day	A.16.3
	ISO 4940:1985 - Flame atomic absorption spectrometric method	r, R	Two to five determinations at a level One determination at a day	A.16.4
S	ISO 4935:1989 - Infrared absorption method after combustion in an induction furnace	r, Rw, R	Three determinations at a level2)	A.20.1
Si	ISO 4829-1:1986 - Reduced molybdosilicate spectrophotometric method - Part 1: Silicon contents between 0,05 and 1,0 %	r, R	Two replications of two independent determinations at a level	A.22.1
	ISO 4829-2:1988 - Reduced molybdosilicate spectrophotometric method - Part 2: Silicon contents between 0,01 and 0,05 %	r, Rw, R	Three determinations at a level2)	___*)
Ti	ISO/DIS 10280 - Diantipyrylmethane spectrophotometric method	ditto	ditto	A.25.1
V	ISO 4942:1988 - N-BPHA spectrophotometric method	ditto	ditto	A.26.1
	ISO 4947:1986 - Potentiometric titration method	r, R	Three independent determinations at a level	A.26.2
	ISO 9647:1989 - Flame atomic absorption spectrometric method	r, Rw, R	Three determinations at a level2)	A.26.3

*) see table 1 (page 20)

NOTES

1 Procedure of calculation of precision is in accordance with ISO 5725.

2 Two of three determinations were carried out under repeatability conditions as defined in ISO 5725, i.e. one operator, same apparatus, identical operating conditions (same calibration) and a minimum period of time.

The third determination was carried out at a different day (different time) by the same operator as in the above using the same apparatus with a new calibration.

From the results obtained on day 1 the **repeatability** (r) and the **reproducibility** (R) were calculated using the procedure specified in ISO 5725. From the first result obtained on day 1 and the result obtained on day 2, the **within-laboratory reproducibility** (Rw) was calculated.

Repeatability, r

The value below which the absolute difference between two single test results obtained on identical material by one operator using the same apparatus within a short time interval may be expected to lie with a probability of 95 % in the normal and correct operation of the method;

Reproducibility, R

The value below which the absolute difference between two single test results obtained by two operators working in different laboratories on identical material may be expected to lie with a probability of 95 % in the normal and correct operation of the method;

Within-laboratory reproducibility, Rw

The value below which the absolute difference between two single test results obtained by the same operator working in the same laboratory on identical material on different days with different calibrations may be expected to lie with a probability of 95 % in the normal and correct operation of the method.

3 'Independent' means that each determination is carried out by a different operator and/or at a different day.

UDC 669.1:543

Descriptors: steels, iron, tests, chemical analysis.

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