



Standard Test Methods for Chemical Analysis of Zinc and Zinc Alloys¹

This standard is issued under the fixed designation E 536; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 These test methods cover the chemical analysis of zinc and zinc alloys having chemical compositions within the following limits:

Element	Concentration Range, %
Aluminum	0.005 to 4.5
Cadmium	0.001 to 0.5
Copper	0.001 to 1.3
Iron	0.001 to 0.1
Lead	0.001 to 1.6
Magnesium	0.001 to 0.1
Tin	0.001 to 0.1

1.2 These test methods appear as follows:

	Sections
Aluminum by the EDTA Titrimetric Method (0.5 to 4.5 %)	8-15
Aluminum, Cadmium, Copper, Iron, Lead, and Magnesium by the Atomic Absorption Method	16-26

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use. Specific precautionary statements are given in Section 5.*

2. Referenced Documents

2.1 ASTM Standards:

- E 29 Practice for Using Significant Digits in Test Data to Determine Conformance with Specifications²
- E 50 Practices for Apparatus, Reagents, and Safety Precautions for Chemical Analysis of Metals³
- E 55 Practice for Sampling Wrought Nonferrous Metals and Alloys for Determination of Chemical Composition³
- E 60 Practice for Photometric and Spectrophotometric Methods for Chemical Analysis of Metals³
- E 88 Practice for Sampling Nonferrous Metals and Alloys in Cast Form for Determination of Chemical Composition³
- E 173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals³

¹ These test methods are under the jurisdiction of ASTM Committee E-1 on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee E01.05 on Zinc, Tin, Lead, Cadmium, Beryllium, and Other Metals.

Current edition approved May 10, 1998. Published July 1998. Originally published as E 536 – 75. Last previous edition E 536 – 84 (1993)¹.

² *Annual Book of ASTM Standards*, Vol 14.02.

³ *Annual Book of ASTM Standards*, Vol 03.05.

3. Significance and Use

3.1 These test methods for the chemical analysis of zinc metals and alloys are primarily intended as referee methods to test such materials for compliance with compositional specifications. It is assumed that all who use these test methods will be trained analysts capable of performing common laboratory procedures skillfully and safely. It is expected that work will be performed in a properly equipped laboratory.

4. Apparatus, Reagents, and Photometric Practice

4.1 Apparatus and reagents required for each determination are listed in separate sections of each test method. The apparatus, standard solutions, and certain other reagents used in more than one procedure are referred to by number and shall conform to the requirements prescribed in Practices E 50, except that photometers shall conform to the requirements prescribed in Practice E 60.

5. Safety Precautions

5.1 For precautions to be observed in the use of certain reagents in these test methods, refer to Practices E 50.

6. Sampling

6.1 For procedures for sampling the material, refer to Practices E 55 and E 88.

7. Rounding Calculated Values

7.1 Calculated values shall be rounded to the desired number of places as directed in 3.4 to 3.6 of Practice E 29.

8. Interlaboratory Studies

8.1 These test methods have been evaluated in accordance with Practice E 173, unless otherwise noted in the precision section.

ALUMINUM BY THE EDTA TITRIMETRIC METHOD

9. Scope

9.1 This test method covers the determination of aluminum in concentrations from 0.5 to 4.5 %.

10. Summary of Test Method

10.1 After dissolution of the sample in hydrochloric acid, the solution is buffered and disodium (ethylenedinitrilo) tetraacetate (EDTA) is added. The excess EDTA is titrated with standard zinc solution. Sodium fluoride is added to decompose

the aluminum-EDTA complex, and the released EDTA is titrated with standard zinc solution.

11. Interferences

11.1 The elements ordinarily present do not interfere if their concentrations are under the maximum limits shown in 1.1.

12. Apparatus

12.1 *Magnetic Stirrer*, with stirring bar covered with TFE-fluorocarbon.

13. Reagents

13.1 *Bromcresol Green Indicator Solution* (0.4 g/L)—Dissolve 0.04 g of bromcresol green in 6 mL of 0.01 N NaOH solution and dilute to 100 mL.

13.2 *EDTA Solution* (90 g/L)—Dissolve 90.0 g of disodium (ethylenedinitrilo) tetraacetate dihydrate in about 800 mL of warm water. Cool and dilute to 1 L.

13.3 *Methyl Red Indicator Solution* (0.4 g/L)—Reagent No. 119.

13.4 *Sodium Acetate Buffer Solution* (320 g/L)—Dissolve 320 g of sodium acetate trihydrate in about 800 mL of water and filter. Using a pH meter, adjust the pH of the solution to 5.5 ± 0.1 with NaOH solution or acetic acid and dilute to 1 L.

13.5 *Sodium Fluoride Solution (Saturated)*—Dissolve 60 g of sodium fluoride (NaF) in 1 L of boiling water. Cool and filter through a coarse paper. Store in a polyethylene bottle.

13.6 *Xylenol Orange Indicator Solution* (10 g/L)—Dissolve 0.250 g of xylenol orange in 25 mL of water. Do not use a solution that has stood more than 1 month.

13.7 *Zinc Standard Solution* (1 mL = 1.00 mg Al)—Dissolve 2.423 g of zinc metal (purity: 99.99 % min) in 20 mL of HCl. Dilute to 100 mL. Add 3 drops of methyl red solution and neutralize with NH₄OH. Add HCl until the color changes to red. Transfer to a 1-L volumetric flask, dilute to volume, and mix.

14. Procedure

14.1 Select and weigh a sample to the nearest 1 mg, in accordance with the following:

Aluminum, %	Sample Weight, g	Aliquot, mL	EDTA Addition, mL
0.5 to 1.5	10.0	50	165 to 168
1.5 to 2.5	6.0	50	103 to 106
2.5 to 4.5	5.0	40	72 to 75

Transfer the sample to a 400-mL beaker, and cover.

14.2 Add 100 mL of HCl (1+1). Heat until dissolution is complete and boil for 2 to 3 min. If a residue remains, add 1 mL of H₂O₂ and boil the solution for at least 5 min to destroy excess H₂O₂ and expel free chlorine.

NOTE 1—Excess peroxide and free chlorine must be removed to prevent fading of the indicators.

14.3 Transfer the solution to a 200-mL volumetric flask, dilute to volume, and mix.

14.4 Using a pipet, transfer the aliquot specified in 14.1 to a 500-mL wide-mouth Erlenmeyer flask.

14.5 Add the volume of EDTA solution specified in 14.1 and dilute to 200 mL.

NOTE 2—The amount of EDTA added must be sufficient to complex the zinc and aluminum with some excess. The amount of EDTA required is 5.7 mg for each milligram of zinc and 14.0 mg for each milligram of aluminum.

14.6 Add 5 or 6 drops of methyl red solution. Add NH₄OH until the color changes to orange.

14.7 Add 25 mL of sodium acetate buffer solution and boil for 3 to 5 min. Cool in a water bath.

14.8 Add 4 drops of xylenol orange solution and 5 or 6 drops of bromcresol green solution.

14.9 Using a TFE-fluorocarbon-covered stirring bar and a magnetic stirrer, stir the solution while adding standard zinc solution from a 50-mL buret to complex the excess EDTA. Add the solution dropwise as the end point is approached. Continue the titration until the color changes from green to red. Refill the buret.

14.10 Add 25 mL of NaF solution and boil for 3 to 5 min. Cool in a water bath.

14.11 Titrate with standard zinc solution as directed in 14.9 and record the volume to the nearest 0.01 mL.

15. Calculation

15.1 Calculate the percentage of aluminum as follows:

$$\text{Aluminum, \%} = (AB/C) \times 100 \quad (1)$$

where:

A = standard zinc solution used in 14.11, mL,

B = aluminum equivalent of the standard zinc solution, g/mL, and

C = sample represented in the aliquot taken, g.

16. Precision and Bias

16.1 *Precision*—Eight laboratories cooperated in testing this test method and obtained the results summarized in Table 1.

16.2 *Bias*—No information concerning the accuracy of this test method is available because certified reference materials suitable for chemical test methods were not available when the interlaboratory test was performed. The analyst is urged to use an accepted reference material, if available, to determine that the accuracy of results is satisfactory.

ALUMINUM, CADMIUM, COPPER, IRON, LEAD, AND MAGNESIUM BY THE ATOMIC ABSORPTION METHOD

17. Scope

17.1 This test method covers the determination of aluminum in concentrations from 0.002 to 0.5 %, cadmium from 0.001 to 0.5 %, copper from 0.001 to 1.3 %, iron from 0.003 to 0.1 %, lead from 0.002 to 1.6 %, and magnesium from 0.001 to 0.1 %.

18. Summary of Test Method

18.1 A hydrochloric acid solution of the sample is aspirated

TABLE 1 Statistical Information

Test Specimen	Aluminum Found, %	Repeatability (R_1 , E 173)	Reproducibility (R_2 , E 173)
G-3	0.3998	0.0190	0.0254
H-2	4.04	0.052	0.074

into the flame of an atomic absorption spectrophotometer. The absorption of the resonance line energy from the spectrum of each element is measured and compared with that of calibration solutions of the same element. The wavelengths of the spectral lines and other method parameters are tabulated in 21.1 for each element.

19. Concentration Range

19.1 The concentration range for each element must be determined experimentally, because the optimum range will depend upon the individual instrument. If the optimum concentration range and instrument parameters have been determined, proceed in accordance with in Section 25; otherwise, determine the concentration range in accordance with in Section 21.

20. Interferences

20.1 The elements ordinarily present do not interfere if their concentrations are under the maximum limits shown in 1.1.

21. Apparatus

21.1 *Atomic Absorption Spectrophotometer*, equipped with a premix burner, with facilities for using the oxidizer-fuel combinations listed in the following table. Use hollow-cathode lamps operated in accordance with manufacturer's recommendations as sources for the spectral lines. The instrument may be considered suitable for this test method if a concentration range can be found for which the minimum response, calibration variability, and reference variability tabulated in the following table can be met:

Element	Spectral Line, Å	Oxidizer-Fuel	Standard Solution	Minimum Response, Units	Calibration Variability, %	Reference Variability, %
Aluminum	3092	N ₂ O-C ₂ H ₂	"A"	9	3.5	2.0
			"B"	25	1.0	0.5
Cadmium	2288	Air-C ₂ H ₂	...	50	0.8	0.4
Copper	3247	Air-C ₂ H ₂	"A"	15	2.0	0.8
			"B"	50	0.8	0.4
Iron	2483	Air-C ₂ H ₂	"A"	15	2.0	1.0
			"B"	25	1.0	0.5
Lead	2170	Air-C ₂ H ₂	"A"	10	2.0	1.5
			"B"	30	1.0	0.4
Magnesium	2852	N ₂ O-C ₂ H ₂	...	20	1.5	0.8

21.1.1 Prepare the dilute standard solution, reference, and calibration solutions in accordance with Section 23. Refer to the table in 23.1 for suggested initial concentrations.

21.1.2 Prepare the instrument for use in accordance with in 25.1. Measure the instrument response while aspirating the reference solution, the lowest, and the two highest calibration solutions, performing the measurements in accordance with 25.2.2 and 25.2.3.

21.1.3 *Minimum Response*—Calculate the difference between the readings of the two highest of the five equally spaced calibration solutions. This difference must be equal to or greater than the number of scale units specified in the table in 21.1. For purposes of this test method, the scale unit is defined

as one in the least significant digit of the scale reading of the most concentrated calibration solution.

21.1.4 *Curve Linearity*—Calculate the difference between the scale readings of the reference solution and the lowest of the five equally spaced calibration solutions. If necessary, convert this difference and the difference calculated in 21.1.3 to absorbance units. Divide the difference for the highest interval by that for the lowest interval. This ratio must be equal to or greater than 0.70.

21.1.5 If the instrument meets or surpasses the minimum response and curve linearity criteria, the initial concentration range may be considered suitable. In this case, proceed in accordance with 21.1.7; otherwise, proceed as follows:

21.1.6 If the minimum response is not achieved, prepare another dilute standard solution to provide a higher concentration range, and repeat 21.1.1-21.1.4. If the calibration curve does not meet the linearity criterion, prepare another dilute standard solution to provide a lower concentration range, and repeat 21.1.1-21.1.4. If a concentration range cannot be found for which both criteria can be met, do not use this test method until the performance of the apparatus satisfies the requirements.

21.1.7 *Instrument Stability*—Calculate the calibration variability and reference variability as follows:

$$V_c = \frac{100}{\bar{c}} \left(\frac{\sum(c - \bar{c})^2}{n - 1} \right)^{\frac{1}{2}} \quad (2)$$

$$V_o = \frac{100}{\bar{c}} \left(\frac{\sum(o - \bar{o})^2}{n - 1} \right)^{\frac{1}{2}} \quad (3)$$

where:

- V_c = calibration variability,
- \bar{c} = average absorbance value for the highest calibration solution,
- $\sum(c - \bar{c})^2$ = sum of the squares of the n differences between the absorbance readings on the highest calibration solution and their average,
- V_o = reference variability relative to c ,
- $\sum(o - \bar{o})^2$ = sum of the squares of the n differences between the absorbance readings on the reference solution and their average, and
- n = number of readings taken on each solution.

21.1.8 If the variability of the readings of the highest calibration and the reference solutions are not equal to or smaller than the values specified in 21.1, the stability of the instrument must be improved before this test method may be used.

22. Reagents

22.1 *Aluminum, Standard Solution* (1 mL = 1.00 mg Al)—Transfer 1 g of aluminum (purity: 99.95 % min) to a 250-mL beaker, cover, and add 50 mL of HCl (1+1). After the reaction has subsided, add 1 mL of H₂O₂ and boil to complete dissolution. Cool and transfer to a 1-L volumetric flask. Add 50 mL of HCl, dilute to volume, and mix. Store in a polyethylene bottle.

22.2 *Cadmium, Standard Solution* (1 mL = 1 mg Cd)—

Transfer 1 g of cadmium (purity: 99.95 % min) to a 250-mL beaker, cover, and add 25 mL of HNO₃(1+1) and 5 mL of HCl. Boil gently to complete dissolution and to remove oxides of nitrogen. Cool and transfer to a 1-L volumetric flask. Add 50 mL of HCl, dilute to volume, and mix. Store in a polyethylene bottle.

22.3 Copper, Standard Solution (1 mL = 1 mg Cu)—Transfer 1 g of copper (purity: 99.95 % min) to a 250-mL beaker, cover, and add 25 mL of HNO₃(1+1) and 5 mL of HCl. Boil gently to complete dissolution and to remove the oxides of nitrogen. Cool and transfer to a 1-L volumetric flask. Add 50 mL of HCl, dilute to volume, and mix. Store in a polyethylene bottle.

22.4 Iron, Standard Solution (1 mL = 1 mg Fe)—Transfer 1 g of iron (purity: 99.95 % min) to a 250-mL beaker, cover, and add 50 mL of HCl (1+1). Boil gently to complete dissolution. Cool and transfer to a 1-L volumetric flask. Add 50 mL of HCl, dilute to volume, and mix. Store in a polyethylene bottle.

22.5 Lead, Standard Solution (1 mL = 1 mg Pb)—Transfer 1 g of lead (purity: 99.95 % min) to a 250-mL beaker, cover, and add 50 mL of HNO₃(1+1). Boil gently to complete dissolution and to remove oxides of nitrogen. Cool and transfer to a 1-L volumetric flask. Add 50 mL of HNO₃, dilute to volume, and mix. Store in a polyethylene bottle.

22.6 Magnesium, Standard Solution (1 mL = 0.1 mg Mg)—Transfer 0.1 g of magnesium (purity: 99.95 % min) to a 250-mL beaker, cover, and add 25 mL of HCl (1+1). Boil gently to complete dissolution. Cool and transfer to a 1-L volumetric flask. Add 50 mL of HCl, dilute to volume, and mix. Store in a polyethylene bottle.

22.7 Sodium Chloride Solution (100 g/L)—Dissolve 100 g of sodium chloride (NaCl) in 1 L of water.

22.8 Zinc, Purified Stock Solution (1 mL = 0.2 g Zn)—Transfer 200 g of zinc (purity: 99.9 % min) to a 2-L beaker. Add 100 mL of water and 700 mL of HCl in small amounts to control the rate of reaction. Add 1 mL of H₂O₂, and evaporate carefully to a syrupy consistency. Dilute to about 800 mL with water, add 30 g of zinc powder (purity: 99.9 % min), and let it react at least 30 min, stirring vigorously several times during the reaction period. Dilute to 1 L with water and filter into a polyethylene bottle. Discard the residue without washing.

NOTE 3—For the determination of aluminum and magnesium in the lowest 10 % of the ranges listed in 17.1, the zinc used must contain less than 0.0002 % Al and 0.0001 % of Mg for samples that contain less than 0.05 % Al and 0.01 % Mg respectively.

23. Calibration

23.1 Dilute Standard Solution—Using pipets, transfer the volume of the appropriate standard solution to a 100-mL volumetric flask in order to obtain the desired concentration of dilute standard solution. Dilute to volume and mix. Use the following as a guide, unless previous experience has shown a different concentration range to be optimum:

Dilute Standard Solution	Concentration Range, %	Volume, mL	Concentration of Dilute Standard Solution, mg/mL
Aluminum "A"	0.004 to 0.020	5	0.05
Aluminum "B"	0.020 to 0.50	20	0.20
Cadmium	0.001 to 0.50	2	0.020
Copper "A"	0.001 to 0.005	2	0.020
Copper "B"	0.005 to 2.5	10	0.10
Iron "A"	0.001 to 0.005	2	0.020
Iron "B"	0.005 to 0.10	10	0.10
Lead "A"	0.001 to 0.005	2	0.020
Lead "B"	0.005 to 2.5	10	0.10
Magnesium	0.001 to 0.10	4	0.004

23.2 Calibration Solutions—Prepare five calibration solutions for each element to be determined in the range required. Using pipets, transfer 5, 10, 15, 20, and 25-mL portions of the appropriate dilute standard solution to five 100-mL volumetric flasks, and provide another 100-mL volumetric flask for the reference solution described in 23.3. Using pipets, add appropriate quantities of NaCl solution, Zn solution, and HCl solution to ensure that the calibration and reference solutions approximate the composition of the test solution. Dilute to volume and mix. Use the following as a guide:

Element	Concentration Range ^A , %	NaCl Solution, mL	Dilute Standard Solution ^B	Zinc Solution, mL	HCl, mL
Aluminum	0.002 to 0.020	5	"A"	25	10
	0.020 to 0.10	5	"B"	25	10
	0.10 to 0.50	5	"B"	5	...
Cadmium	0.001 to 0.005	...	Cadmium	50	20
	0.005 to 0.025	...	Cadmium	10	5
	0.025 to 0.125	...	Cadmium	2	...
	0.10 to 0.50	...	Cadmium
Copper, Iron, or Lead	0.001 to 0.005	...	"A"	50	20
	0.005 to 0.025	...	"B"	50	20
	0.025 to 0.125	...	"B"	10	5
	0.125 to 0.625	...	"B"	2	...
	0.50 to 2.5	...	"B"
Magnesium	0.001 to 0.005	5	Magnesium	10	5
	0.005 to 0.025	5	Magnesium	2	...
	0.020 to 0.10	5	Magnesium

^A These values are based upon dilute standard solutions prepared in accordance with the values in 23.1. If higher or lower concentrations are used for the dilute standards, the concentration ranges covered by the calibration solution will be changed accordingly.

^B The "A" or "B" designates the A or B dilute standard solution of the appropriate element.

23.3 Reference Solution—Prepare a reference solution containing the same volumes of reagents and zinc solution as the calibration solutions but without addition of the dilute standard solution.

24. Procedure

24.1 Test Solution:

24.1.1 Transfer a 10-g sample, weighed to the nearest 10 mg, to a 400-mL beaker, except as follows: use a 2.5-g sample for determining copper and lead in concentrations greater than 0.5 %. Cover and add 50 mL of HCl in small increments to control the rate of reaction. Heat gently to complete the dissolution. Add 0.5 mL of H₂O₂ and boil gently for 5 min. Cool, transfer to a 100-mL volumetric flask, dilute to volume, and mix.

NOTE 4—For aluminum in the range from 0.002 to 0.020 % only, add 5 mL of NaCl solution before diluting to volume and omit 24.1.2.

24.1.2 Using a pipet, transfer an appropriate volume of the sample solution to a 100-mL volumetric flask, dilute to volume, and mix:

Element	Concentration Range, %	Sample Solution, mL/100 mL	NaCl Solution, mL
Aluminum	0.002 to 0.020	100 ^A	5
	0.020 to 0.10	50	5
	0.10 to 0.50	10	5
Cadmium	0.001 to 0.005	100 ^B	...
	0.005 to 0.025	20	...
	0.025 to 0.125	4	...
	0.10 to 0.50	1	...
Copper, Iron, and Lead	0.001 to 0.005	100 ^B	...
	0.005 to 0.025	100	...
	0.025 to 0.125	20	...
	0.125 to 0.625	4	...
	0.50 to 1.6 ^C	4	...
Magnesium	0.001 to 0.005	20	5
	0.005 to 0.025	4	5
	0.020 to 0.10	1	5

^A See Note 3.

^B The test solution is the undiluted sample solution.

^C For lead and copper above 0.5 %, use a 2.5-g sample.

25. Measurement

25.1 Instrument Adjustment:

25.1.1 Set the instrument parameters approximately to those known to be optimum for the element to be determined. Light the burner.

25.1.2 Adjust the instrument to the approximate wavelength for the element to be determined and aspirate water until thermal equilibrium is established. Aspirate the highest calibration solution, and adjust the wavelength to obtain maximum response.

25.1.3 Optimize fuel, air, and burner adjustments while aspirating the highest calibration solution.

25.1.4 Aspirate water long enough to establish that the instrument reading is stable, and then set the initial reading (approximately zero absorbance, or 100 % transmittance). Verify that the reference solution gives readings that are above zero absorbance (less than 100 % transmittance).

25.2 Photometry:

25.2.1 Aspirate the test solution and note, but do not record the reading.

25.2.2 Aspirate water until the initial reading is again obtained. Aspirate the reference, calibration, and test solutions

TABLE 2 Statistical Information

Specimen	Average, %	Repeatability, (R ₁ , E173)	Reproducibility, (R ₂ , E173)
Al	G-1	0.0015	0.0006
	G-3	0.401	0.035
Cd	H-2	0.0008	0.0003
	G-5	0.545	0.025
Cu	G-3	0.0015	0.0003
	H-2	1.154	0.09
Fe	G-1	0.0026	0.0006
	H-3	0.113	0.012
Pb	H-2	0.0016	0.0003
	G-5	1.368	0.12
Mg	G-3	0.0010	0.0003
	H-2	0.055	0.009

in order of increasing instrument response. Record the reading when a stable response is obtained from each solution.

25.2.3 Proceed as directed in 25.2.2 at least two more times to obtain a minimum of three readings for each solution.

26. Calculation

26.1 Calculate the variability of the readings for the highest calibration and reference solutions as directed in 21.1.7. If the calculated values are not equal to or less than the maximum values given in 21.1 for the element being determined, disregard the data, readjust the instrument, and proceed again in accordance with 25.2.

26.2 If necessary, convert the average of the readings of each of the calibration solutions and the test solution to absorbance.

26.3 Prepare a calibration curve by plotting the absorbance values for the calibration solutions against milligrams of the element per 100 millilitres.

26.4 Convert the absorbance value of the test solution to milligrams by means of the calibration curve.

26.5 Calculate the percentage of the element as follows:

$$\text{Element, \%} = \frac{A}{B} \times 100 \tag{4}$$

where:

- A = element in 100 mL of the test solution, mg, and
- B = sample in 100 mL of the test solution, mg.

27. Precision and Bias ⁴

27.1 Precision—Six laboratories cooperated in testing this test method and obtained eight sets of data which are summarized in Table 2.

27.2 Bias—No information on the accuracy of this test method is available because no certified reference materials suitable for chemical test methods were available when the interlaboratory test was performed. The analyst is urged to use a certified reference material, if available, to determine that the accuracy of results is satisfactory.

28. Keywords

28.1 aluminum; cadmium; copper; iron; lead; magnesium; zinc; zinc alloys

⁴ Supporting data are available from ASTM Headquarters. Request RR:E03-1010.

 **E 536**

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