SRI LANKA STANDARD 300 : 1986

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SPECIFICATION FOR
CAUSTIC SODA (TECHNICAL GRADES)
(FIRST REVISION)

SRI LANKA STANDARDS INSTITUTION

SPECIFICATION FOR CAUSTIC SODA (TECHNICAL GRADES)

(FIRST REVISION)

SLS 300:1986
(Attached AMD 141)

Gr. 8

SRI LANKA STANDARDS INSTITUTION
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SRI LANKA STANDARD
SPECIFICATION FOR CAUSTIC SODA (TECHNICAL GRADES)
(FIRST REVISION)

FOREWORD

This Sri Lanka Standard was authorized for adoption and publication by the Council of the Sri Lanka Standards Institution on 1986-07-18, after the draft, finalized by the Drafting Committee on Chemicals and Chemical Products, had been approved by the Chemicals Divisional Committee.

This specification was first published in 1974. In this revision requirements for 73 per cent *(m/m)* solution of caustic soda have been deleted since it is not used in the country. Changes have been made in the requirements for chlorate content of caustic soda in both solution form and solid form and for iron content, copper content and matter insoluble in water content in caustic soda solution. New test methods have been introduced for the determination of carbonate, iron and copper. Method of sampling has also been modified.

All values given in this specification are in SI units.

For the purpose of deciding whether a particular requirement of this specification is complied with, the final value, observed or calculated, expressing the results of a test or analysis shall be rounded off in accordance with CS 102. The number of significant places retained in the rounded off value shall be the same as that of the specified value in this specification.

In the preparation of this specification, valuable assistance derived from the publications of the International Organization for Standardization, the British Standards Institution and the Indian Standards Institution is gratefully acknowledged.

1 SCOPE

1.1 This specification prescribes requirements, methods of sampling and tests for caustic soda used in soap, textile, paper and other industries not requiring a special grade of the material. It covers the material in the solid form and solution.

1.2 It does not specify the requirements of caustic soda intended for medicinal use, photographic use and for cosmetic industry.

2 REFERENCES

ISO	979	Sodium hydroxide for industrial use - method of assay
ISO	982	Sodium hydroxide for industrial use - determination of sulfate content - barium sulfate gravimetric method
ISO	983	Sodium hydroxide for industrial use - determination of iron content - 1, 10-phenanthroline photometric method
ISO	3195	Sodium hydroxide for industrial use - sampling - test sample - preparation of the main solution for carrying out certain determinations
ISO	3196	Sodium hydroxide for industrial use - determination of carbonates content - titrimetric method
CS	102	Presentation of numerical values
SLS	428	Random sampling methods
SLS	692	Safety colours and safety signs

3 REQUIREMENTS

3.1 General requirements

3.1.1 The material shall be supplied in either solid form or solution form.

3.1.2 The material shall be free from odour, dirt, foreign matter, and other visible impurities.

3.1.3 The solid caustic soda shall essentially consist of sodium hydroxide. It shall be a deliquescent solid in the form required by the purchaser, for example : fused material, flake, pearl, powder, stick or pellets.

3.1.4 The solution of caustic soda shall essentially consist of sodium hydroxide in water.

3.2 Other requirements

Caustic soda shall conform to the requirements given in Table 1, when tested according to the methods given in Column 5 of the table. The requirement for maximum iron content shall not apply to fused material solidified in the drum.

TABLE 1 - Requirements for caustic soda

Sl. No. (1)	**Characteristic** (2)	**Requirement**		**Method of test** (5)
		Solid (3)	**Solution** (4)	
i)	Caustic alkalinity, as NaOH, per cent by mass, min.	95.0	46.0	**ISO 979**
ii)	Carbonate, as CO_2, per cent by mass, max.	0.8	0.2	**ISO 3196**
iii)	Sulfate, as Na_2SO_4, per cent by mass, max.	0.05	0.05	**ISO 982**
iv)	Iron, as Fe, mg/kg, max.	40	100	**ISO 983**
v)	Chloride, as NaCl, per cent by mass, max.	2.5	1.3	**Appendix B**
vi)	Chlorate, as $NaClO_3$, per cent by mass, max.	0.005	1.0	**Appendix C**
vii)	Copper, as Cu, mg/kg, max.	2.0	1.0	**Appendix D**
viii)	Matter insoluble in water, per cent by mass, max.	0.05	Not specified	**Appendix E**

4 PACKAGING AND MARKING

4.1 The solid caustic soda shall be supplied in clean, securely closed containers. Each container shall be legibly and indelibly marked or labelled with the following information :

a) Name and address of the manufacturer and/or distributor (including the country of origin);

b) Registered trade mark, if any;

c) The words *CAUSTIC SODA (NaOH), TECHNICAL*;

d) Percentage of sodium hydroxide;

e) Mass of the material in the container, in kilograms;

f) Pictorial marking for *Corrosive material*, (see warning sign B.3.4 given in **SLS 692**) ; and

g) Pictorial marking for *This way up* (see Fig. 1).

4.2 The caustic soda solution shall be delivered in clean, bulk containers.

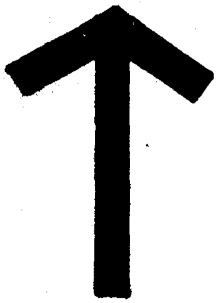

FIGURE 1 - Pictorial marking for "This way up"

4.3 Each container may also be marked with the Certification Mark of the Sri Lanka Standards Institution, illustrated below on permission being granted for such marking by the Sri Lanka Standards Institution.

NOTE - The use of the Sri Lanka Standards Institution Certification Mark (SLS Mark) is governed by the provisions of the Sri Lanka Standards Institution Act and the regulations framed thereunder. The SLS Mark on products covered by a Sri Lanka Standard is an assurance that they have been produced to comply with the requirements of that standard under a well defined system of inspection, testing and quality control, which is devised and supervised by the Institution and operated by the producer. SLS marked products are also continuously checked by the Institution for conformity to the relevant standard as a further safeguard. Details of conditions under which a permit for the use of the Certification Mark may be granted to manufacturers or processors may be obtained from the Sri Lanka Standards Institution.

5 SAMPLING

The method of drawing representative samples of the material for ascertaining conformity to the requirements of this specification shall be as prescribed in Appendix A.

6 METHODS OF TEST

6.1 During the analysis, unless otherwise stated, use only reagents of recognized analytical grade and only distilled water or water of equivalent purity.

6.2 Tests for the requirements laid down in **3**, shall be carried out as prescribed in **ISO 982**, **ISO 983**, **ISO 979**, **ISO 3196** and **Appendices B** to **E** of this specification. Preparation of the main solution for carrying out some of these tests shall be as prescribed in **ISO 3195**.

7 CONFORMITY TO STANDARD

A lot shall be declared as conforming to the requirements of this specification if the following conditions are satisfied :

7.1 Each container inspected as in **A.5.1** satisfies the packaging and marking requirements.

7.2 The test results of the composite sample when tested as in **A.5.2** satisfy the relevant requirements.

APPENDIX A

SAMPLING

A.1 GENERAL REQUIREMENTS OF SAMPLING

A.1.1 Precautions shall be taken to protect samples, the material being sampled, the sampling instrument and the containers for samples from adventitious contamination, particularly from absorption of moisture and carbon dioxide.

A.1.2 The samples shall be placed in clean, dry and air-tight alkali resistant glass containers.

A.1.3 Sampling instrument shall be cleaned before use.

A.1.4 To draw representative samples, the contents of each container selected for sampling shall be mixed thoroughly by suitable means.

A.1.5 Each sample container shall be sealed air-tight after filling and marked with necessary details of sampling.

A.2 LOT

In a single consignment, all containers of the product manufactured under the same conditions of manufacture or belonging to one batch of supply shall constitute a lot.

A.3 SCALE OF SAMPLING

A.3.1 Samples shall be taken from each lot for ascertaining conformity of the material to the requirements of this specification.

A.3.2 Selection of samples

A.3.2.1 *Solid caustic soda*

A.3.2.1.1 The number of containers to be selected from a lot shall be in accordance with Table 2.

TABLE 2 - Scale of sampling

Number of containers in the lot	**Number of containers to be selected**
Up to 50	2
51 to 90	3
91 to 150	4
151 to 280	5
281 to 500	8
501 and above	10

A.3.2.1.2 The containers shall be selected at random. In order to ensure randomness of selection tables of random numbers as given in **SLS 428** shall be used.

A.3.2.2 *Caustic soda solution*

Representative samples of the material shall be drawn from each bulk container using an appropriate sampling instrument.

A.4 PREPARATION OF SAMPLES

A.4.1 Solid caustic soda

Representative samples of the material shall be drawn from each container selected as in **A.3.2** using an appropriate sampling instrument. The material taken from each container shall be mixed to form a composite sample of not less than 300 g and shall be transferred to a clean and thoroughly dried sample container.

A.4.2 Caustic soda solution

The samples drawn as in **A.3.2.2** shall be mixed to form a composite sample of not less than 200 ml.

A.5 NUMBER OF TESTS

A.5.1 Each container selected as in **A.3.2.1** shall be inspected for packaging and marking requirements (This may be done at the place of inspection).

A.5.2 The composite sample prepared as in **A.4.1** or **A.4.2** shall be tested for all requirements of this specification.

APPENDIX B

DETERMINATION OF CHLORIDE

B.1 PRINCIPLE

Precipitation of chloride ions by addition of excess silver nitrate and titration of the excess with ammonium thiocyanate in the presence of ammonium ferric sulfate.

B.2 REAGENTS AND MATERIALS

B.2.1 *Concentrated nitric acid*, rel. den. = 1.40

B.2.2 *Litmus paper*

B.2.3 *Ammonium ferric sulfate, nitric acid solution.* Dissolve 25 g of ammonium ferric sulfate hydrate $(NH_4)_2SO_4.Fe_2(SO_4)_3.24H_2O$ in 100 ml of water. Add 50 ml of concentrated nitric acid solution (rel. den. = 1.40), and mix thoroughly.

B.2.4 *Silver nitrate*, standard volumetric solution, $c(AgNO_3)$ = 0.1 mol/l (see Note).

B.2.5 *Ammonium thiocyanate*, standard volumetric solution. $c(NH_4SCN)$ = 0.1 mol/l (see Note).

B.2.6 *Nitric acid*, approximately 0.2 mol/l solution.

NOTE - If the silver nitrate (**B.2.4**) *and ammonium thiocyanate* (**B.2.5**) *standard volumetric solution are not exactly the strength indicated in the list of reagents, suitable correction factors shall be employed in calculating the results.*

B.3 APPARATUS

Ordinary laboratory apparatus.

B.4 PROCEDURE

Transfer 100.0 ml of sample Solution A (see **ISO 3195**) to a 500-ml conical flask. Neutralize this, with nitric acid solution **(B.2.1)** in the presence of litmus paper **(B.2.2)**. Then add 0.5 ml to 1 ml excess nitric acid solution **(B.2.1)** and cool under running water to room temperature. Add approximately 3 ml of the ammonium ferric sulfate solution. **(B.2.3)**.

Fill a burette with silver nitrate solution **(B.2.4)** and another with ammonium thiocyanate solution **(B.2.5)**. Run 0.20 ml of the latter solution into the sample solution so as to form ferric thiocyanate, a red compound, the appearance of which indicates the end-point of the titration. Then add silver nitrate solution **(B.2.4)** until the reddish-pink colour disappears and add an excess of approximately 2 ml measured to the nearest 0.05 ml. Stir for 4 to 5 minutes and filter under reduced pressure on a galss filter crucible fitted with a sintered disk of porosity between 5 mm and 15 mm. Wash both the conical flask and the filter crucible three times with a small amount (approximately 10 ml) of nitric acid solution **(B.2.6)** and then titrate the excess silver nitrate in the filtrate by adding ammonium thiocyanate solution **(B.2.5)** until the pink colour reappears. Deduct from the volume of the titrant, the drop that causes the end-point.

B.5 CALCULATION

Chloride content, as NaCl, per cent by mass

$$= (V_o - V_1) \times m_1 \times \frac{1\ 000}{100} \times \frac{100}{m_o}$$

$$= 5.844 \times \frac{(V_o - V_1)}{m_o}$$

where,

V_o = volume, in millilitres, of the silver nitrate standard volumetric solution used;

V_1 = volume, in millilitres, of the ammonium thiocyanate standard volumetric solution used, (0.20 ml + volume used for back titration);

m_o = mass, in grams, of the test portion used for the preparation of the sample solution A; and

m_1 = mass, in grams, of sodium chloride corresponding (theoretical value 1 ml = 0.005 844 g of sodium chloride) to 1 ml of 0.1 mol/l silver nitrate solution.

APPENDIX C

DETERMINATION OF CHLORATE

C.1 PRINCIPLE

Treatment with concentrated acid and passing evolved chlorine into excess of potassium iodide solution. Titration of liberated iodine with standard sodium thiosulfate solution.

C.2 REAGENTS AND MATERIALS

C.2.1 *Piece of marble,* 5 g to 10 g.

C.2.2 *Potassium iodide solution.* Dissolve 2.5 g of potassium iodide in 42 ml of water.

C.2.3 *Concentrated hydrochloric acid,* rel. den. = 1.18

C.2.4 *Sodium thiosulfate,* standard volumetric solution. $c(Na_2S_2O_3)$ = 0.1 mol/l.

C.2.5 *Starch,* 10 g/l solution.

C.3 APPARATUS

C.3.1 *Ordinary laboratory apparatus*

C.3.2 *100-*ml *round bottom quickfit flask*

C.3.3 *Two U-tubes,* connected as shown in Figure 2 and immersed in a bath of cold water.

C.3.4 *Quickfit delivery tube*

C.4 PROCEDURE

Weigh, to the nearest 0.01 g, a mass of the sample corresponding to 25 g of sodium hydroxide and transfer into a flask **(C.3.2)** and to this add a small piece of marble **(C.2.1)**. Introduce 34 ml of potassium iodide solution **(C.2.2)** into the first U-tube and 8 ml into the second U-tube and connect the two together. Add 20 ml of hydrochloric acid **(C.2.3)** to the content of the flask and connect it by means of a delivery tube **(C.3.4)** to the first U-tube. Heat the mixture very gently so that the chlorine is slowly evolved. Increase the temperature gradually to the boiling point; the carbon dioxide evolved expels all the chlorine, and this liberates the iodine from the potassium iodide solution. Immediately before the heating is stopped, disconnect the U-tubes from the delivery tube in order to prevent the solution in the absorption vessels from passing back into the flask. Transfer the contents of both U-tubes to a conical flask and titrate the liberated iodine at once with sodium thiosulfate **(C.2.4)** until the solution is pale yellow in colour. Add 2 ml of starch solution **(C.2.5)** and continue the addition of the thiosulfate solution slowly until the solution is just colourless.

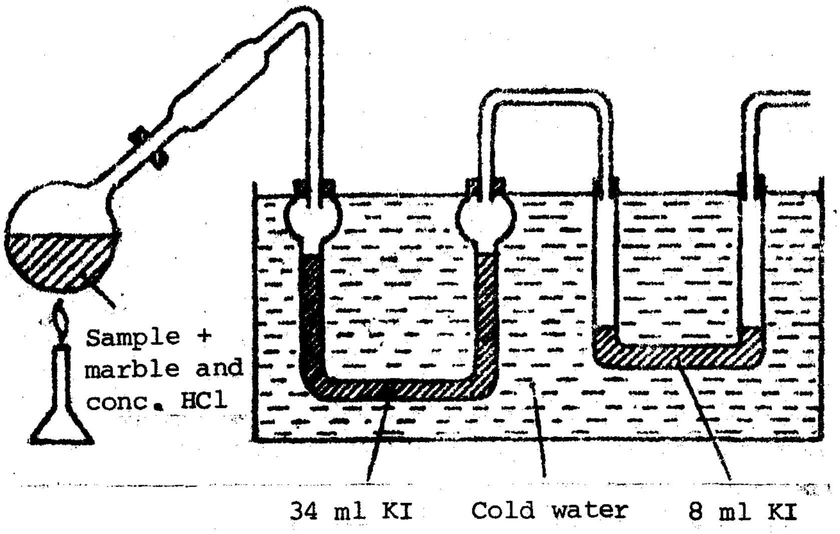

FIGURE 2 - Apparatus for determination of chlorate

C.5 CALCULATION

Chlorate content, as $NaClO_3$, per cent by mass

$$= V \times \frac{106.448}{1\,000} \times \frac{0.2}{6} \times \frac{100}{m}$$

$$= 0.354\,83 \times \frac{V}{m}$$

where,

V = volume, in millilitres, of sodium thiosulfate standard volumetric solution used; and

m = mass, in grams, of the test sample used.

APPENDIX D

DETERMINATION OF COPPER

D.1 APPARATUS

D.1.1 *Ordinary laboratory apparatus*

D.1.2 *Nessler cylinders,* 100-ml capacity

D.2 REAGENTS AND MATERIALS

D.2.1 *Concentrated sulfuric acid,* rel. den. = 1.84

D.2.2 *Litmus paper*

D.2.3 *Ammonium chloride*

D.2.4 *Concentrated ammonium hydroxide,* rel. den. = 0.92

D.2.5 *Citric acid solution.* Dissolve 10.0 g of citric acid (monohydrate) crystals in 100 ml of water.

D.2.6 *Dilute ammonium hydroxide,* approximately 3 mol/l

D.2.7 *pH papers*

D.2.8 *Gum acacia suspension.* Dissolve 1.0 g of gum acacia in 100 ml of boiling water, filter and dilute to 200 ml. (This solution shall be prepared fresh.)

D.2.9 *Tetrasodium pyrophosphate solution.* Dissolve 4 g of tetrasodium pyrophosphate in 100 ml of water.

D.2.10 *Sodium diethyl dithiocarbamate solution.* Dissolve 0.2 g of sodium diethyl dithiocarbamate in water and make up the volume to 100 ml.

D.2.11 *Standard copper solution.* Dissolve 0.392 g of copper sulfate pentahydrate ($CuSO_4.5H_2O$) in slightly acidic water and make up the volume to 1 litre in a volumetric flask. Pipette out exactly 10 ml of the solution in a 100-ml volumetric flask and make up the volume to 100-ml mark. One millilitre of this solution is equivalent to 0.01 mg of copper.

D.3 PROCEDURE

D.3.1 Weigh, approximately 10 g of the material, to the nearest 0.1 g, and transfer it into a 400-ml beaker. Dissolve it in about 20 ml of water and then neutralize with concentrated sulfuric acid **(D.2.1)** using litmus paper **(D.2.2)**. To the neutralized solution add ammonium chloride **(D.2.3)** and concentrated ammonium hydroxide **(D.2.4)**

to precipitate out iron. Heat the solution to coagulate the precipitate. Filter the precipitate through a sintered crucible with an asbestos mat and wash the residue twice with water. Collect the filtrate and the washing in a beaker. Cover the beaker with a watch glass and then evaporate to a volume of about 70 ml. Transfer the solution to a Nessler cylinder **(D.1.2)**. To this add 2 ml of citric acid solution **(D.2.5)** and add dilute ammonium hydroxide **(D.2.6)** checking the pH value using pH papers **(D.2.7)**, until pH = 8.5.

Mix the solution thoroughly and then add 10 ml of gum acacia suspension **(D.2.8)**, 5 ml of tetrasodium pyrophosphate solution **(D.2.9)** and 10 ml of sodium diethyl dithiocarbamate solution **(D.2.10)** and dilute to 100-ml mark with distilled water.

To another Nessler cylinder **(D.1.2)** add 2 ml of citric acid solution **(D.2.5)**, dilute ammonium hydroxide **(D.2.6)** until pH = 8.5, 10 ml of gum acacia suspension **(D.2.8)**, 5 ml of tetrasodium pyrophosphate solution **(D.2.9)** 10 ml of soidum diethyl dithiocarbamate solution **(D.2.10)** and then 2 ml of standard copper solution **(D.2.11)** when testing the solid caustic soda, or 1 ml of standard copper solution when testing the solution of caustic soda. Make up to 100-ml mark with distilled water and mix well. Compare the colour produced in the two cylinders after 10 minutes.

D.3.2 The limit prescribed shall be taken as not being exceeded if the intensity of the colour produced in the test with the material is not greater than that in the control test.

APPENDIX E

DETERMINATION OF MATTER INSOLUBLE IN WATER

E.1 PRINCIPLE

Dissolution of a test portion. Filtration of the solution through a tared filter crucible. Weighing of the water-insoluble residue.

E.2 APPARATUS

E.2.1 *Ordinary laboratory apparatus*

E.2.2 *Sintered glass or porous porcelain crucible*, having porosity between 5 µm and 15 µm, which has been pre-treated as follows:

Filter 200 ml of approximately 100 g/l sodium hydroxide solution through the crucible. Rinse thoroughly with water to ensure that any loose particles and all the residual sodium hydroxide solution is removed. Dry in an oven **(E.2.3)** controlled within the temperature range of 100 °C to 105 °C, for one hour and allow to cool in a desiccator.

NOTE - The crucible may be used for subsequent determinations without further pretreatment.

L.2.3 *Oven*, capable of being controlled within the temperature range of 100 °C to 105 °C.

E.3 PROCEDURE

Weigh, to the nearest 0.1 g, in a plastic weighing bottle fitted with a cover, about 50 g of the sample. Transfer to a 400-ml beaker and add about 250 ml of water. Cool the solution to room temperature and then filter through a clean dry crucible **(E.2.2)** previously weighed to the nearest 0.1 mg, transferring any insoluble matter into the crucible with a jet of water. Wash the filter five times with 5-ml portions of water at room temperature. Dry the crucible and contents for one hour in an oven **(E.2.3)** at 100 °C to 105 °C, cool in a desiccator and weigh to the nearest 0.1 mg.

E.4 CALCULATION

$$\text{Matter insoluble in water, per cent by mass} = \frac{m_1}{m_o} \times 100$$

where,

m_o = mass, in grams, of the test portion; and

m_1 = mass, in grams, of the matter insoluble in water.

AMENDMENT NO. 1 TO SLS 300 : 1986
SPECIFICATION FOR CAUSTIC SODA (TECHNICAL GRADE)
(1ST REVISION)

EXPLANATORY NOTE

Paranthan Chemicals Company Limited, imports the full requirement of caustic soda for the entire country for paper, soap and textile industry. It has been experienced that very few foreign suppliers are able to supply this material conforming to the maximum copper content specified in the present Sri Lanka Standard Specification.

The above Sri Lanka Standard specifies copper, as Cu, 2.0 mg/kg maximum for solid and 1.0 mg/kg maximum for solution. According to most of the analytical data available, the copper level of caustic soda (Tech. Grade) exceeds the maximum limit specified in the standard. It has also been found in chemical catalogs available, maximum limit of 5 mg/kg copper content for sodium hydroxide analytical grade.

The Institution contacted relevant industries using caustic soda. It was observed that for the soap industry, where the copper content is important a limit of 5 mg/kg would be acceptable. For other industries a maximum limit of 5 mg/kg is quite adequate.

This amendment is introduced to change the maximum limit for copper content from 2.0 mg/kg to 5 mg/kg for solid caustic soda.

Amendment No. 1 approved on 1992-03-26
to SLS 300 : 1986
SPECIFICATION FOR CAUSTIC SODA (TECHNICAL GRADE)
(FIRST REVISION)

Page 5

In Table 1 Sl. No. vii, column 3 delete "2.0" and substitute "5".

SRI LANKA STANDARDS INSTITUTION

The Sri Lanka Standards Institution (SLSI) is the national standards organization of Sri Lanka established by the Sri Lanka Standards Institution Act No. 6 of 1984 which repeals the Bureau of Ceylon Standards Act No. 38 of 1964. The Institution functions under the Ministry of Industries and Scientific Affairs.

The principal objects of the Institution as set out in the Act are to prepare standards and promote their adoption, to provide facilities for examination and testing of products, to operate a Certification Marks Scheme, to certify the quality of products meant for local consumption or exports and to promote standardization and quality control by educational, consultancy and research activity.

The Institution is financed by Government grants, and by the income from the sale of its publications and from other services. Financial and administrative control is vested in a Council appointed in accordance with the provisions of the Act.

The detailed preparation of standard specifications is done by Drafting Committees composed of experts in each particular field assisted by permanent officers of the Institution. These Committees are appointed by the Divisional Committees, which in turn are appointed by the Council. All members of the Drafting and Divisional Committees render their services in an honorary capacity. In preparing the standard specifications, the Institution endeavours to ensure adequate representation of all view points.

In the international field the Institution represents Sri Lanka in the International Organization for Standardization (ISO), and participates in such fields of standardization as are of special interest to Sri Lanka.

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