3 Mannington Ct
St. Peters, Missouri 63376
(636) 447-1312
(636) 614-2599 FAX

Laszlo Corporation

Technical Specification		
	Caprolactam	WT-2001/ZA-5/angl instead of WT-99/ZA-5/angl

Approved on 6 December 2001, by

1. Introduction

1.1 Subject

The subject of this Technical Specification is caprolactam, $C_6H_{11}ON$, manufactured from benzene, sold in liquid form or as solid flakes.

1.2 Scope of application

Caprolactam is used for the manufacture of polyamide polymer which is subsequently processed into textile/technical yarn and engineering plastics used for the manufacture of a wide variety of engineering and industrial goods.

2. Designation

Caprolactam WT-2001/ZA-5/angl

3. Requirements

3.1 General requirements

Liquid caprolactam is colorless, clear, free of any suspended matter. Solid caprolactam is white, crystalline, with a slight, specific smell.

3.2 Detailed requirements according to Table 1

Table 1

No.	Requirements	Units	Value
1.	Permanganate number, at least	s	20000
2.	Volatile bases content, max	mmol/kg	0.3
3.	Water content, max	%	0.05
4.	Crystallizing point, at least	°C	69.0
5.	Color of 50% aqueous solution at 390 nm, max	Hazen units	3
6.	Alkalinity, max	mmol/kg	0.05
7.	Acidity, max	mmol/kg	0.05
8.	Absorbance of 50% aqueous solution, max	-	0.04
9.	Iron content, max	mg/kg	0.5
10.	Ash content, max	mg/kg	5
11.	Cyclohexanone oxime content, max	mg/kg	2

4. Packing, storage and transportation

4.1 Packing

Liquid caprolactam is loaded into road tankers or railway tank cars made of stainless steel; the contact zones must be free of any colored seals and color non-ferrous metals, as these could color caprolactam. Metal parts and seals in contact with caprolactam must not change the quality of caprolactam and must be resistant to it, e.g. stainless steel. As the volatile bases build up when liquid caprolactam is in contact with air, an oxygen-free blanket of slightly pressurized nitrogen must be maintained over liquid caprolactam. Solid caprolactam is loaded into 25 kg PE bags closed by welding. The PE bags with caprolactam are stacked on pallets and wrapped up with STRETCH film, are packed into double bags containing as an external bag a PP-reinforced PE bag or a PP-reinforced paper bag, closed by sewing. The internal bag is not connected with the external bag. The double bags are basically intended for manual handling, however, they can also be stacked on pallets and protected by wrapping up with STRETCH film. The net weight of caprolactam on a single pallet is up to 1500 kg.

4.2 *Storage*

Liquid caprolactam shall be stored in stainless steel tanks, thermally insulated and heated with hot water. Caprolactam temperature should be maintained at 80-90°C and may not exceed 95°C. Liquid caprolactam must be stored under an oxygen-free, slightly pressurized nitrogen blanket.

Solid caprolactam shall be stored in original packaging, i.e. on pallets covered with film and wrapped up with STRETCH film, or, alternately, in double bags, at storage room temperatures no more than 45°C. and moderate air humidity. Caprolactam may not be exposed to direct sunlight.

4.3 *Transportation*

Liquid caprolactam is transported in road tankers or railway tank cars intended for caprolactam service, made of stainless steel, provided with thermal insulation, tank heating system and piping.

If necessary, road tankers and tank cars may be heated with low-pressure steam before unloading. Solid caprolactam is transported in containers or weather-protected wagons.

5. Testing

5.1 Testing schedule according to Table 2

Table 2

No.	Test Requirements	Test description according to:	according to:
1.	Checking for general requirements	3.1	5.4.1
2.	Determination of permanganate number	3.2.1	5.4.2
3.	Determination of volatile bases content	3.2.2	5.4.3
4.	Determination of water content	3.2.3	5.4.4
5.	Determination of crystallizing point	3.2.4	5.4.5
6.	Determination of color of 50% aqueous solution	3.2.5	5.4.6
7.	Determination of alkalinity	3.2.6	5.4.7
8.	Determination of acidity	3.2.7	5.4.8
9.	Determination of absorbance of 50% aqueous solution	3.2.8	5.4.9
10.	Determination of iron content	3.2.9	5.4.10
11.	Determination of ash content	3.2.10	5.4.11
12.	Determination of cyclohexanone oxime content	3.2.11	5.4.12

5.2 *Lot size*

For liquid caprolactam. a lot is the content of a single tank car to be shipped. For solid caprolactam, a batch/lot is an amount of 16-25 tons of final product that can be loaded into a single container. A shipment lot is a single consignment of up to 500 tons to be sent to a customer under a single shipment order.

5.3 *Sampling and preparation of final bulk sample*

Sampling shall be made in accordance with PN-C-04333: 2000. During sampling, one must pay attention to the fact that caprolactam is hygroscopic and that volatile bases build-up occurs in contact with oxygen. Sampling, especially for the liquid caprolactam, should be performed so as to avoid any increase of the content of moisture and/or other impurities in the sample taken.

Liquid caprolactam: A sample of liquid caprolactam is split into a test sample for immediate analysis and an archive sample to be kept, properly marked, for 3 months from the date of shipment. The archive sample of caprolactam is kept in the solid form.

Solid caprolactam: From a batch of caprolactam, a bulk sample of at least 1 kg is taken from caprolactam packing line. After mixing, the sample is split into two parts: one for immediate analysis and the other one as archive sample. A final bulk sample of a consignment is

prepared from samples of individual batches by mixing 1/3 parts of archive samples from individual batches. The consignment final bulk sample is split into two parts: one for immediate analysis and the other one as archive sample. Archive samples of each individual batch and the consignment final bulk sample are stored for 6 months from the date of shipment.

5.4 *Testing description*

5.4.1 Checking for general requirements

Transfer liquid caprolactam into a polyamide bag made from a sleeve and compare its appearance with that of water in a PA bag as above. Place a solid caprolactam sample in a clear glass cylinder and check its appearance.

5.4.2 Determination of permanganate number: according to PN-93/C-45301/03.

5.4.3 Determination of volatile bases content: according to PN-93/C-45301/06 or ISO 8661-1988.

5.4.4 Determination of water content: according to PN-81/C-04959.

5.4.5 Determination of crystallizing point: according to PN-93/C-45301/08 or ISO 7060-1982.

5.4.6 Determination of color of 50% aqueous solution at 390 nm: according to PN-93/C-45301/04 or ISO 8112-1984.

5.4.7 Determination of alkalinity: according to PN-93/C-45301/09

5.4.8 Determination of acidity: according to PN-93/C-45301/09

5.4.9 Determination of absorbance of 50% aqueous solution: according to PN-45301/05 or ISO 7059-1982

5.4.10 Determination of iron content: according to PN-93/C-45301/10

5.4.11 Determination of ash content

5.4.11.1 Principle

The method consists in decomposition of caprolactam by incineration, roasting at 800°C and weighing the residue. 5.4.11.2 Apparatus

a) platinum crucible, approx. 100 ml,

b) furnace with a temperature range up to 1000°C,

c) desiccator,

d) beaker,

e) burner.

5.4.11.3 Determination procedure

Hold the platinum crucible in the furnace at 800°C for 30 minutes, then remove it from the furnace and place in the desiccator. After the crucible cools down to room temperature, weigh it with an accuracy of 0.0001 g (G_1).

Weigh 500 g of caprolactam into the beaker. Transfer approximately 50 g of caprolactam from the beaker into the platinum crucible and carefully melt it over the flame of a gas burner. Continue heating until the caprolactam ignites, then remove the burner flame and allow the substance to burn out completely. Repeat this operation 9 times (take approx. 50 g of caprolactam each time), until total 500 g of caprolactam is completely incinerated. Next, hold the crucible for 40 minutes in the furnace at 800°C so that all carbonized products are burned out. Subsequently, place the crucible in the desiccator and, after it cools down to room temperature, weigh with an accuracy of 0.0001 g.

Next, place the crucible again in the furnace at 800°C. After 20 minutes, place the crucible in the desiccator and, after it cools down to room temperature, weigh it with an accuracy of 0.0001 g (G2).

Ash content (x) in the caprolactam shall be calculated in mg/kg according to the following formula:

$$x=(G_2-G_1) \cdot 2 \cdot 1000$$

where: G_1 = weight of platinum crucible alone, in grams

G_2 = weight of platinum crucible with residue, in grams 5.4.11.4 Final result

The final result shall be an arithmetic mean from the results of at least 2 parallel determinations, differing not more than 3 mg/kg.

5.4.12 Determination of oxime content

5.4.12.1 Principle

The method consists in hydrolyzing cyclohexanone oxime to cyclohexanone and hydroxylamine, oxidation of hydroxylamine with iodine to nitric acid which will form a colored compound in a reaction with sulphanilic acid and a-naphthylamine. Absorbance of the solution tested, measured at 520 nm wavelength, is proportional to the content of cyclohexanone oxime. 5.4.12.2 Reagents and materials

Only analytical grade reagents and distilled water are allowed for the analysis.

a) concentrated hydrochloric acid, d = 1.19,

b) glacial acetic acid, c = 99.5% wt.

c) sulphanilic acid at c = 1% wt., prepared as follows:

Dissolve 10 g sulphalinic acid in 750 ml distilled water and 250 ml glacial acetic acid, heat that mixture until it boils and a homogenous liquid is formed. Store the thus obtained solution in a dark glass bottle.

d) iodine solution at c(I) =0.1 mol/l, prepared as follows:

Dissolve 12,7 g iodine in a sodium iodide solution (16 g sodium iodide in 125 ml distilled water) in a measuring flask of 1 liter capacity, and make up to the mark with distilled water. Store in a dark glass bottle.

e) sodium acetate (CHaCOONa • 3 H2O) solution prepared as follows: Dissolve 225 g sodium acetate in distilled water in a 1 l measuring flask, make up to the mark with distilled water and mix.

f) sodium thiosulfate solution at c(Na2S203) =0.1 mol/l,

g) a-naphthylamine solution at c = 0.3% wt., prepared as follows: Dissolve 3.0g a-naphthylamine in a solution of acetic acid (300 ml glacial acetic acid mixed with 750 ml distilled water). Store the solution in a dark glass bottle for a period of time no longer than 1 week. h) caprolactam free of cyclohexanone oxime, i) cyclohexanone oxime. 5.4.12.3 Apparatus

a) spectrometer with a range covering 520 nm wavelength, b) two absorption cells having optical path of 5 cm, c) hydrolysis set consisting of a round, flat-bottom 500 ml flask with a reflux condenser

(ground-in glass connectors). 5.4.12.4 Determination procedure

5.4.12.4.1 Preparation of a scale of standards and a standard curve

Prepare cyclohexanone oxime standard solution as follows:

Solution 1: Dissolve 0.5000 g cyclohexanone oxime in distilled water in 500 ml measuring flasks. Make up to the mark with distilled water and mix (1 ml solution contains 1 mg cyclohexanone oxime).

Solution 2: Place 50 ml of Solution 1 in a 500 ml measuring flask. Make up to the mark with distilled water and mix (1 ml solution contains 0.1 ml cyclohexanone oxime).

Solution 3: Place 50 ml of Solution 2 in a 500 ml measuring flask. Make up to the mark with distilled water and mix (1 ml solution contains 0.01 ml cyclohexanone oxime). Place in subsequent 500 ml round-bottom flasks the following amounts: 0, 5.0, 10.0, 20.0, 30.0, and 50.0 ml of Solution 3, which corresponds, respectively, to 0, 1, 2, 4, 6, and 10 mg/kg cyclohexanone oxime in caprolactam. Add distilled water to each flask so that total solution volumes are 100 ml each (100, 95, 90, 80, 70, and 50 ml). Add 50 g caprolactam free of cyclohexanone oxime to each flask.

Further, proceed according to 5.4.12.4.2, starting from adding 37,5 ml hydrochloric acid. Plot a standard curve with the results obtained, with absorbance values on Y axis and the content of cyclohexanone oxime in caprolactam in mg/kg on X axis.

5.4.12.4.2 Cyclohexanone oxime in caprolactam determination procedure

Weigh 50 g caprolactam at an accuracy of 0.01 g and transfer that amount quantitatively into a 500 ml round-bottom flask. Add 100 ml of distilled water and 37.5 ml hydrochloric acid (d == 1.19). Connect the flask with a reflux condenser and keep the solution boiling for 1 hour. Cool down the solution to room temperature. Transfer the solution quantitatively into a 250 ml measuring flask. Flush the condenser and the flask with 60 ml of distilled water; then add that water to the solution. Make up to the mark with water and mix. Transfer 25 ml of that solution into a 100 ml measuring flask. Add 20 ml of sulphanilic acid solution (ace. to 5.4.12.2-c), 1 ml iodine solution (ace. to 5.4.12.2.d) and mix. Put the flask in a dark place. After 15 minutes, add 20 ml sodium acetate solution (ace. to 5.4.12.2.e), 2 ml sodium thiosulfate (ace. to 5.4.12.2.f) and 10 ml a-naphthylamine solution (ace. to 5.4.12.2.g). Mix the content. Make up to the mark with water and mix. Place the flask in a dark place; after 30 minutes measure absorbance at 520 nm wavelength in 5 cm optical path absorption cells, against a blank sample (containing caprolactam free of cyclohexanone oxime). Read the content of cyclohexanone oxime in caprolactam, in mg/kg, from the standard curve prepared according to 5.4.12.4.1.

5.4.12.5 Final result

The final result shall be an arithmetic mean of the results of at least 2 parallel determinations, differing no more than 2 mg/kg.

5.5 *Evaluation of results and quality control certificate*

A batch of caprolactam shall be deemed to comply with the requirements of this Technical Specification when the test results meet the requirements provided in Table 1 herein. The manufacturer is obliged to send to the customer a certificate stating that the product complies with the requirements of this Technical Specification.

THE END

Additional information to WT-2001/ZA-5/angl

1) Organization that has developed this Technical Specification:
2) Related standards and other documents

- PN-C 04333: 2000 - Coal downstream products. Sampling and preparation of a final bulk sample.
- PN-81/C-04959 - Determination of water content in organic and inorganic products by Karol Fischer method.
- PN-93/C-45301/01 - Caprolactam. Methods of testing. General provisions and scope of the standard.

PN-93/C-45301/02 - Caprolactam. Methods of testing. Spectrometric determination of permanganate indicator.

- PN-93/C-45301/03 - Caprolactam. Methods of testing. Visual determination of permanganate number of a 3% aqueous solution.
- PN-93/C-45301/04 - Caprolactam. Methods of testing. Spectrometric determination of color, in Hazen units, of a 50% aqueous caprolactam solution.
- PN-93/C-45301/05 - Caprolactam. Methods of testing. Determination of absorbance at 290 nm.
- PN-93/C-45301/06 - Caprolactam. Methods of testing. Determination of volatile bases content.
- PN-93/C-45301/08 - Caprolactam. Methods of testing. Determination of crystallizing point.
- PN-93/C-45301/09 - Caprolactam. Methods of testing. Determination of acidity or alkalinity.
- PN-93/C-45301/10 - Caprolactam. Methods of testing. Determination of iron content.
- PN-85/0-79252 - Transportation containers with content. Marks and marking. Basic requirements.
- ISO 7059-1982 - Caprolactam for industrial use - Determination of absorbance at wavelength of 290 nm.
- ISO 7060-1982 - Caprolactam for industrial use - Determination of crystallizing point.
- ISO 8112-1984 - Caprolactam for industrial use - Determination of color of 50% aqueous caprolactam solution expressed in Hazen units (platinum-cobalt scale). Spectrometric method.
- ISO 8661-1988 - Caprolactam for industrial use - Determination of volatile bases content. Titrimetric method after distillation.

Additional information to WT-2001/ZA-5/angl

1) Organization that has developed this Technical Specification:
2) Related standards and other documents

- PN-C 04333: 2000 - Coal downstream products. Sampling and preparation of a final bulk sample.
- PN-81/C-04959 - Determination of water content in organic and inorganic products by Karol Fischer method.
- PN-93/C-45301/01 - Caprolactam. Methods of testing. General provisions and scope of the standard.

PN-93/C-45301/02 - Caprolactam. Methods of testing. Spectrometric determination of permanganate indicator.

- PN-93/C-45301/03 - Caprolactam. Methods of testing. Visual determination of permanganate number of a 3% aqueous solution.
- PN-93/C-45301/04 - Caprolactam. Methods of testing. Spectrometric determination of color, in Hazen units, of a 50% aqueous caprolactam solution.
- PN-93/C-45301/05 - Caprolactam. Methods of testing. Determination of absorbance at 290 nm.
- PN-93/C-45301/06 - Caprolactam. Methods of testing. Determination of volatile bases content.
- PN-93/C-45301/08 - Caprolactam. Methods of testing. Determination of crystallizing point.
- PN-93/C-45301/09 - Caprolactam. Methods of testing. Determination of acidity or alkalinity.
- PN-93/C-45301/10 - Caprolactam. Methods of testing. Determination of iron content.
- PN-85/0-79252 - Transportation containers with content. Marks and marking. Basic requirements.
- ISO 7059-1982 - Caprolactam for industrial use - Determination of absorbance at wavelength of 290 nm.
- ISO 7060-1982 - Caprolactam for industrial use - Determination of crystallizing point.
- ISO 8112-1984 - Caprolactam for industrial use - Determination of color of 50% aqueous caprolactam solution expressed in Hazen units (platinum-cobalt scale). Spectrometric method.
- ISO 8661-1988 - Caprolactam for industrial use - Determination of volatile bases content. Titrimetric method after distillation.

- Order by Minister of Transportation, dated 6 October 1987, concerning a list of dangerous goods excluded from railway transportation, and special conditions for the transportation of the dangerous goods allowed for transportation item 169, year 1987).
- Order by Minister of Transportation and Internal Affairs, dated 2 December 1983, concerning conditions and monitoring of transportation of dangerous materials tem 301, year 1983, and No. 42, item 206, year 1986) Rules for International Railway Transportation (COTIF), Appendix B, unified regulations on international railway transportation (CIM), Annex 1: Rules for International Raj|way Transportation of Dangerous Goods (RID)
- European Agreement concerning the international road transportation of dangerous goods (ADR).