

To
NEDEX KIMYA SAN. VE.TIC.A.S.
Sokak No:1 Elite Plaza
34775 Umraniye (ISTANBUL)
TURKIYE

Att.: Mr. Aslan Bilmen

Marghera, February 26th, 2015

Object: prEN 1279-4: October 2014 tests on ZEOLAN K molecular sieves

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1 Charge definition

Stazione Sperimentale del Vetro has been in charge of the execution of tests according to prEN 1279-4 October 2014 version on desiccant Zeolan K produced by Nedex.

2 Reference

The client sent to Stazione Sperimentale del Vetro n.1 box of desiccant Zeolan K, on December 11th, 2014.

3 Tests

The following tests have been performed on the desiccant.

3.1 Loss of Ignition (LOI)

LOI is determined, checking the weight difference before and after thermic treatment at 540°C.

Test has been executed according to annex E.1.

A porcelain crucible is employed: "m₀" is the registered weight of the empty crucible; "m₂" is the initial weight after filling with approximately 25 grams of salt. A crucible containing desiccant has been treated during at least two hours at 540°C and then inserted in a glass desiccator to cool at room temperature. This weight correspond to "m₁".

LOI is determined as follows:

$$\text{LOI} = \frac{m_2 - m_1}{m_1 - m_0} \cdot 100$$

3.2 AWAC

Test for of available water adsorption capacity of desiccant salts (AWAC), has been performed according to annex E.2.

A fraction of desiccant was placed in a glass container. Then the recipient containing the salt was introduced in a desiccator. In the bottom of this one a KOH saturated solution having a temperature of 23°C ± 3°C was present. In these conditions, the relative humidity of air, over the saturated solution surface, will be 9% at equilibrium state. The quantity of salt present in the desiccator has not to be more than a 0.3 g/l.

The desiccator as prepared was closed during 72 hours, then opened and the weight of the recipient containing the salt was checked.

Each draft was registered (initial "W0", after salt introduction "W1" after 72 hours "W2").

At least water adsorption capacity of salt by means of the following formula was calculated:

$$\text{AWAC} = \frac{W2 - W1}{W1 - W0} \cdot 100$$

3.3 Tc determination

As indicated in annex E.3 of prEN 1279-4 October 2014 version, Tc is determined adding AWAC and LOI values.

3.4 Grain Size analysis

The tests were performed using a set of ISO3310-1 stainless steel sieves with apertures varying from 0.1 mm to 1 mm.

3.5 X-Ray Fluorescence Spectroscopy (XRF) (according to point 6.2.1 of prEN 1279-4:October 2014)

The analyses were performed on the powdered samples after calcinations at 1050 °C, using a sequential X-ray Spectrometer calibrated with certified standard reference materials of rocks, mineral, glasses and high purity oxides.

The results are reported in table 5 as oxides weight %.

3.6 X-Ray Diffraction (XRD) (according to point 6.2.2 of prEN 1279-4:October 2014)

The analyses were performed on the powdered samples, using a X-ray diffractometer.

3.7 Gas desorption

Test has been executed according to annex E.4 of prEN 1279-4: October 2014.

Samples has been kept at room temperature conditions.

The desiccant (without having done any specific thermal treatment) has been inserted in a 250 ml container; then the container has been connected by means of a tube to a graduate cylinder pre-filled with water and placed upside down inside a recipient containing 1 liter of water:

The recipient containing the desiccant is immersed in water at a temperature of 70°C:

- After 60 minute it is measured the gas volume that left to the cylinder after desiccant heating; following measurement has been performed each 30 minutes during 4 hours (total time).

If the difference between measurement n.1 and measurement n.2 is > 0.9 this result is defined as “instable”.

- desorpted gas volume (in ml/g) is defined using the last value registered after 4 hours by the following formula:

$$\text{desorpted gas} = \text{final volume of desorpted gas} / \text{desiccant mass}$$

Tests has been repeated 3 times for each desiccant typology.

4 Results

4.1 LOI

Table 1: L.O.I. Verification Results

| LOI% test n°1 | LOI% test n°2 | LOI% test n°3 | Average value LOI % |
|------------------|------------------|------------------|------------------------|
| 0.3 | 0.4 | 0.3 | 0.3 |

4.2 AWAC

Table 2: Water absorbtion capability verification results

| AWAC% test n°1 | AWAC% test n°2 | AWAC% test n°3 | Average value AWAC% |
|-------------------|-------------------|-------------------|------------------------|
| 13.8 | 13.8 | 14.0 | 13.9 |

4.3 Tc determination

Table 3: Tc determination

| LOI+AWAC | Tc % |
|------------|-------------|
| (0.3+13.9) | 14.2 |

4.4 Grain Size analysis

Table 4: Grain size distribution

| Sieve (mm) | Mass fraction (wt. %) |
|------------|-----------------------|
| > 1.0 | 0,00 |
| 0.8 - 1.0 | 24.22 |
| 0.71 - 0.8 | 49.81 |
| 0.6 - 0.71 | 24.87 |
| 0.5 - 0.6 | 0.66 |
| 0.4 - 0.5 | 0.15 |
| 0.3 - 0.4 | 0.11 |
| 0.2 - 0.3 | 0.08 |
| 0.1 - 0.2 | 0.08 |
| < 0.1 | 0.02 |

4.5 X-Ray Fluorescence Spectroscopy (XRF)

Table 5: Quantitative chemical composition

| <i>Wt %</i> | ZEOLAN K |
|------------------------------------|----------|
| SiO ₂ | 51.4 |
| Al ₂ O ₃ | 27.5 |
| Na ₂ O | 7.6 |
| K ₂ O | 7.7 |
| CaO | 2.2 |
| MgO | 1.6 |
| SO ₃ tot | 0.09 |
| Fe ₂ O ₃ tot | 1.8 |
| TiO ₂ | 0.26 |
| P ₂ O ₅ | 0.09 |

4.6 X-Ray Diffraction (XRD)

The analyses were performed on the powdered samples, using a X-ray diffractometer. Results are summarized in table 6. The XRD patterns are reported in fig. 1

Table 6: *Results of phase analyses (XRD)*

| SAMPLE | PHASE |
|----------|--|
| ZEOLAN K | zeolite A quartz - SiO ₂ |

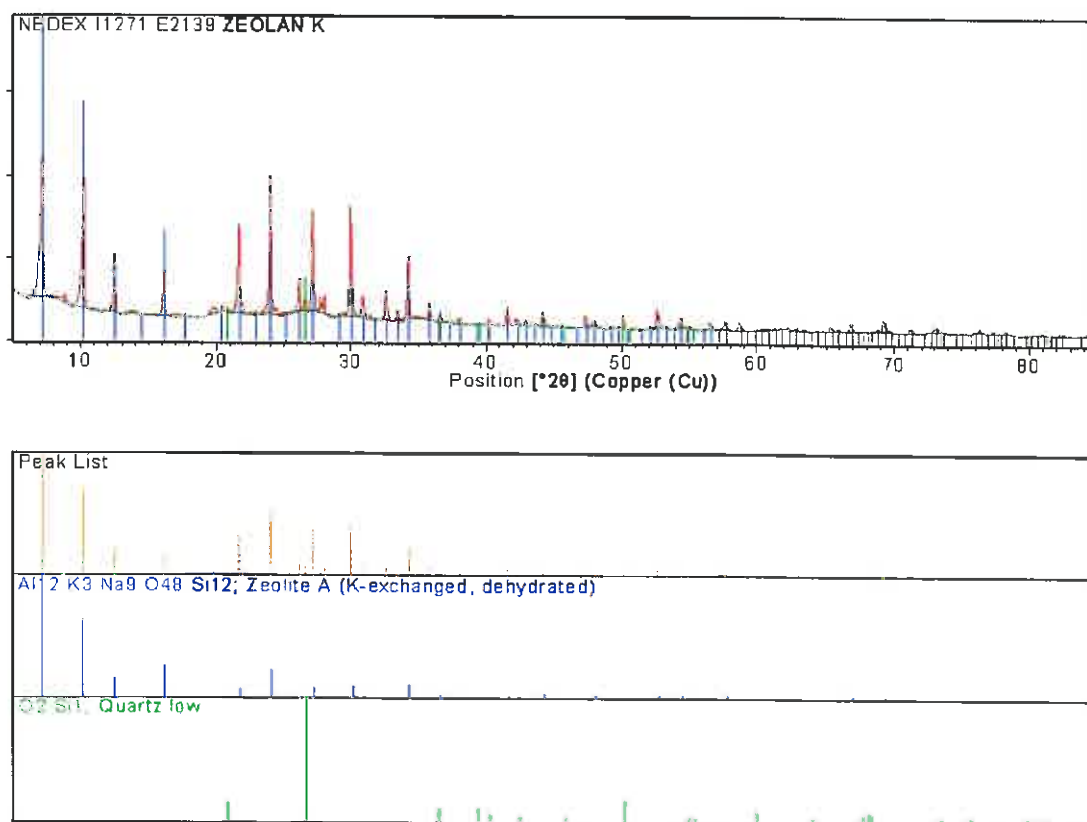


Fig.1 X-Ray Diffraction pattern

4.7 Gas desorption

Table 7: Verifying of gas desorption

| Test n°1 ml/g | Test n°2 ml/g | Test n°3 ml/g | Average value ml/g |
|------------------|------------------|------------------|-----------------------|
| 0.38 | 0.32 | 0.31 | 0.34 |

Tests 4.1 4.2 4.3 and 4.7 has been executed Marghera labs.

Tests 4.4 4.5 and 4.6 has been executed at Murano labs.

THE HEAD OF THE LABORATORY

Ennio Mognato

THE LABORATORIES DIRECTOR

Dr. Nicola Favaro

Stazione Sperimentale del Vetro S.c.p.A. - The Glass Research Center

Via Briati, 10 - 30141 Murano (VE) • P.I. 04176390278 • T.+39 041.2737011 • F.+39 041.2737048 • www.spevetro.it • mail@spevetro.it
Sede secondaria: c/o VEGA Edificio Pegaso - Via delle industrie, 13 - 30175 Venezia Marghera • T. +39 041.5383112 • F. +39 041.5090669