Ammonium, nitrate, and total nitrogen determination in fly ash

KjelMaster K-375, SpeedDigester K-439, Scrubber K-415, DuMaster D-480

Fly ash is a by-product of the combustion of pulverized coal and is composed of small particles. The composition of the fly ash may vary considerably, however, all fly ashes contain substantial amounts of silicon dioxide (SiO_2), aluminum oxide (Al_2O_3) and calcium oxide (CaO). Nowadays, air pollution control standards require a separation of flue gas before being released to the atmosphere. Fly ash, incurred in the flue gas can be re-used e.g. as fill material or raw material for cement [1].

The fly ash sample analyzed in this study was provided from a pulp mill containing organic and inorganic (ammonium nitrate and ammonium sulfate) nitrogen compounds.

1. Introduction

The aim of this application note was to determine the amount of different nitogen compounds in fly ash. A clever workflow, applying the KjelMaster K-375, allows the selective determination of the total organic nitrogen, i.e. mainly glycine, as well as the inorganic ammonium and nitrate.

Complementary, the total nitrogen content, of organic and inorganic nature, was analyzed by applying the DuMaster D-480. An overview about the nitrogen compounds which can be analyzed with the two different methods, are shown in figure 1.

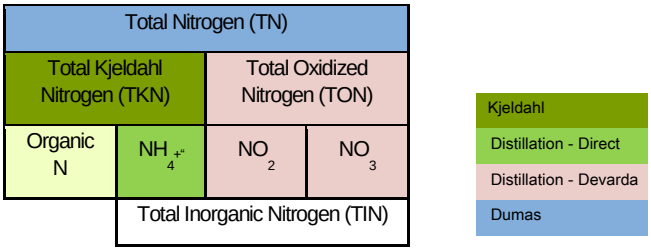

Figure 1: Nitrogen compounds which can be analyzed with the two methods Kjeldahl and DuMaster.

2. Experimental

To determine the **total Kjeldahl nitrogen** (TKN), the fly ash sample was digested, then, the distillation and boric acid titration were performed. Importantly, if the sample contains ammonium, the organic nitrogen and the ammonium are determined as TKN. To quantify the organic nitrogen amount only, the ammonia content was measured in addition and subtracted from the total Kjeldahl nitrogen content.

The **ammonium** (NH_4^+) only, was quantified by alkalization of the sample followed by direct steam distillation and boric acid titration.

The **nitrate** (NO_3) content was determined after reduction to ammonia using Devarda's alloy and subsequent steam distillation [2]. To save time and sample, the already distilled sample of the ammonium determination was reused for nitrate determination.

The **total nitrogen** (TN) was measured according to Dumas using the DuMaster D-480 and compared to the sum of the nitrogen containing compounds (organic nitrogen, ammonia and nitrate) measured with the KjelMaster K-375.

All determinations were performed four fold (n = 4).

3. Results

For the total Kjeldahl nitrogen the mean value was 6.26 % (0.84 % Rsd). As the fly ash sample contains also ammonium of inorganic origin, e.g. ammonium nitrate, both, ammonia from organic and inorganic origin are co-distilled according to the Kjeldahl method.

The ammonium from inorganic origin, determined with distillation only, was 4.76 % N (0.84 % Rsd). Hence, the difference between total Kjeldahl nitrogen (6.26 % N) and directly distilled ammonium (4.76 % N) is the organic nitrogen (1.50 % N).

The nitrate determination (Devarda method) was performed after the ammonium distillation. The amount of nitrogen obtained from nitrate was 2.63 % (1.3 % Rsd).

The sum of the nitrogen from organic origin (1.50 %), ammonium (4.76 %), and nitrate (2.63 %) results in the total nitrogen using Kjeldahl apparatus (8.90 % N).

The total nitrogen according to the Dumas method (9.66 % N) was higher than the Kjeldahl result (+ 0.76 %) indicating the presence of further inorganic nitrogen sources in the sample. Figure 2 shows an overview of the obtained and calculated results.

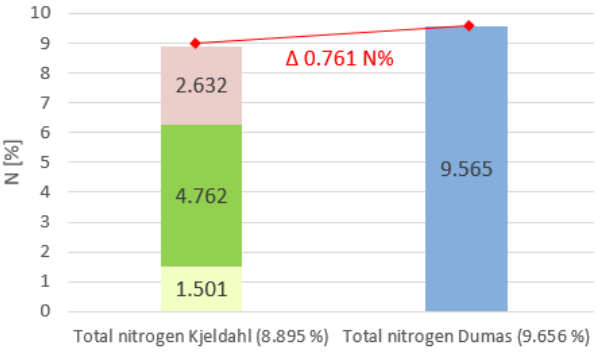

Figure 2: Summary of the nitrogen content measured in fly ash.

4. Conclusion

Using the K-375 and applying a smart workflow, fly ash is analyzed for its nitrogenous compounds. Ammonium from organic and inorganic origin can be discriminated, nitrate exactly determined, and the total nitrogen content is analyzed.

Comparison of the found total nitrogen content to the nitrogen contend obtained using the DuMaster D-480 clearly show that additional inorganic compounds are expected in the analyzed sample.

5. References

[1] http://flyash.com/about-fly-ash/

[2] Application Note No. 116 / 2013; Nitrogen determination in sodium nitrate

For more detailed information and safety conside-rations please refer to the Application Note No. 235/2016.