Contamination-free sample preparation and transport of granular chips used for combustion analysis

Abstract

Herzog recently introduced a novel technology enabling the production of granular chips especially suited for thermal evolution analysis. In this application note we investigated whether the automatic collection and transport of granular chips may cause contamination from chips that have become stuck throughout the pneumatic tube line. For this purpose, we examined the impact of inserting a sample with high C and S content on the analytical outcome in a series of samples with low C and S content. In ten milling cycles with a total of 80 trials, we found no evidence of significant contamination (student's t-test, $P<0.05$). The reverse case, insertion of a low-concentration sample into a sample series with a high C and S content, also did not reveal any contamination. This shows that the fully-automatic process of chip production, collection and transport results in reliable analytical results.

Key words

• **Combustion analysis** • **Chips** • **Milling** • **Contamination** • **Automation**

Introduction

The cylindrical milling cutter module within the HS-F 1000 milling machine enables the production of short chips having a uniform and even morphology. In the previous application notes we demonstrated that the thermal evolution analysis of the so-called "granular" chips resulted in an excellent repeatability [1]. Furthermore, we showed that the circumferential scale layer of production samples did not have any significant impact on C and S content as measured by combustion analysis [2].

Both studies were conducted using granular chips collected from a cup inside the HS-F 1000 and then manually processed by the operator. For fully-automatic sample preparation and analysis by, e.g., using the Herzog CNSLab, the collected chips must be automatically transported to a dosing unit. One option is to use a pneumatic transport system that connects the chip production module inside the HS-F 1000 with the dosing unit in the CNSLab.

In this application note we aim at investigating whether the pneumatic transport causes any contamination of the chips by the preceding sample. For this purpose, we examined the influence of inserting a sample with high C and

S content on the analysis of subsequent samples with low C and S content. Additionally, we evaluated whether, conversely, a sample with low C and S content may change the analytical values of samples with high C and S content.

Methods

The chips were produced in an automatic milling machine of the type HS-F 1000 with an integrated module for production of granular chips. All samples were manually inserted into the machine. For each preparation cycle, the top layer of the sample was first removed by a face-milling cutter. Afterwards, the support of the HS-F 1000 transported the sample to the chip production module. Here, the sample was passed over a cylindrical milling cutter with 30 cutting tips.

The chips were collected by an integrated crescent-shaped trap and transported from there pneumatically via a pipeline to a cyclone. There, the chips were separated and collected in a cup positioned below in the receiving station of the CNSLab (Figure 1). All components of the pneumatic transport system were designed to prevent chips from getting stuck. This was achieved, among other things, by constructively avoiding gaps and dead spaces and adjusting the internal contour to achieve optimal airflow.

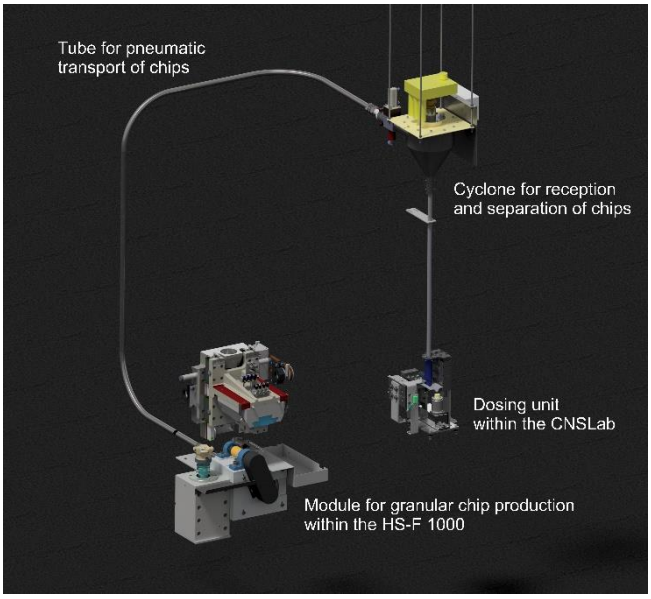

Figure 1: *Basic experimental setup with chip transport from the HS-F 1000 to the receiving cup in the dosing unit of the CNSLab.*

For this test series we omitted automatic dosing in the CNSLab and instead prepared the chips manually for the thermal evolution analysis. For determination of C and S, we weighed 500-1000 mg of the chips into a ceramic crucible according to the sample type to be analyzed. Subsequently, we added 1.5- 2.0 g tungsten as accelerator and introduced the crucible into the analyzer (Elementrac CS-i, Eltra, Haan Germany). As more chips were available in the receiving cup than were necessary for the combustion we took care to select a representative analytical sample.

In the first test series we examined whether insertion of a sample with high C and S content (C60 sample) caused contamination of chip samples with low C and S content (RH 31/32). Hence, we first carried out two tests in which chips from the RH 31/32 sample were prepared and analyzed. In trial 3, chips were prepared from the C60 sample and transported to the dosing unit. Subsequently, five more trials were carried out with preparation and transport of RH31/32 samples (Figure 2 A). The purpose was to determine whether the preparation of the C60 sample led to contamination of the subsequent RH31/32 chip samples and, accordingly, to an increase in C and S content. We conducted 10 preparation cycles with eight trials each leading to a total sum of 80 trials.

In the second test series we assessed whether the RH 31/32 sample influenced the C and S content of a series of C60 samples. Accordingly, the sample order was reversed from that in the first part of the study (Figure 2 B). Also for this test series a total sum of 80 trials was conducted.

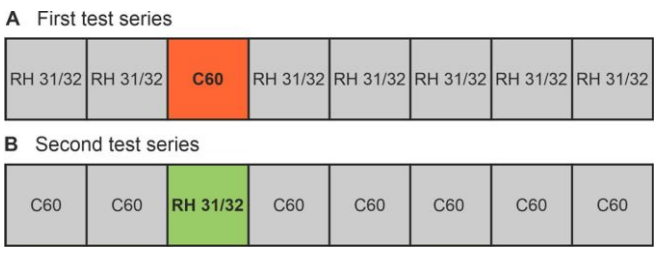

Figure 2: *Experimental design of contamination tests: (A) In trial 3, a C60 sample was inserted into a series of RH 31/32 samples , (B) in trial 3, a RH 31/32 sample was inserted into a series of C60 samples.*

We calculated the mean and standard deviation from each set of ten trials. We then used student's t-tests ($P< 0.05$) to examine whether the C and S content measured in each trial was statistically different from each other.

Results

1. Impact of insertion of a C60 sample into a series of RH31/32 samples

In the first two trials the mean C content (± standard deviation) was 0.0336 ± 0.0020 % and 0.0360 ± 0.0043 %, respectively (Table 1, Figure 3). In trial 3 the C60 sample was inserted leading to an increase of the mean C content to 0.7535 ± 0.0141 %. In the subsequent five trials (again preparation of RH31/32 samples) the C content was between 0.0323 ± 0.0009 % and 0.0356 ± 0.0031 %. Student's t-test revealed that only trial 3 was significantly different from other trials (t-test group B, Table 1). All other trials using RH31/32 samples showed no significant differences (t-test group A, Table 1). In particular, no increase in C content was observed in the trials after inserting the C60 sample. The mean S content after preparation of RH31/32 samples was between 0.0116 ± 0.0004 % and 0.0127 ± 0.0022 % (Table 1, Figure 3).

Insertion of the C60 sample (trial 3) led to an increase of the mean S content to 0.0363 ± 0.0016 %. Whereas student's t-test revealed no difference of the S content between RH31/32 samples (t-test group A, Table 1) the only significant increase was observed after insertion of the C60 sample (t-test group B).

Trial	Carbon			Sulfur		
	Mean ± SD	t-test group		Mean ± SD	t-test group	
1	0.0336 ± 0.0020	A		0.0116 ± 0.0004	A	
2	0.0360 ± 0.0043	A		0.0121 ± 0.0010	A	
3	0.7535 ± 0.0141		B	0.0363 ± 0.0016		B
4	0.0356 ± 0.0031	A		0.0125 ± 0.0012	A	
5	0.0333 ± 0.0020	A		0.0122 ± 0.0006	A	
6	0.0339 ± 0.0022	A		0.0127 ± 0.0022	A	
7	0.0336 ± 0.0020	A		0.0121 ± 0.0017	A	
8	0.0323 ± 0.0009	A		0.0119 ± 0.0012	A	

Table 1: *Mean percentage content ± standard deviation for C and S in each of the eight trials of the first test series. Trials which share the same test-group character showed no significant difference (t-test).*

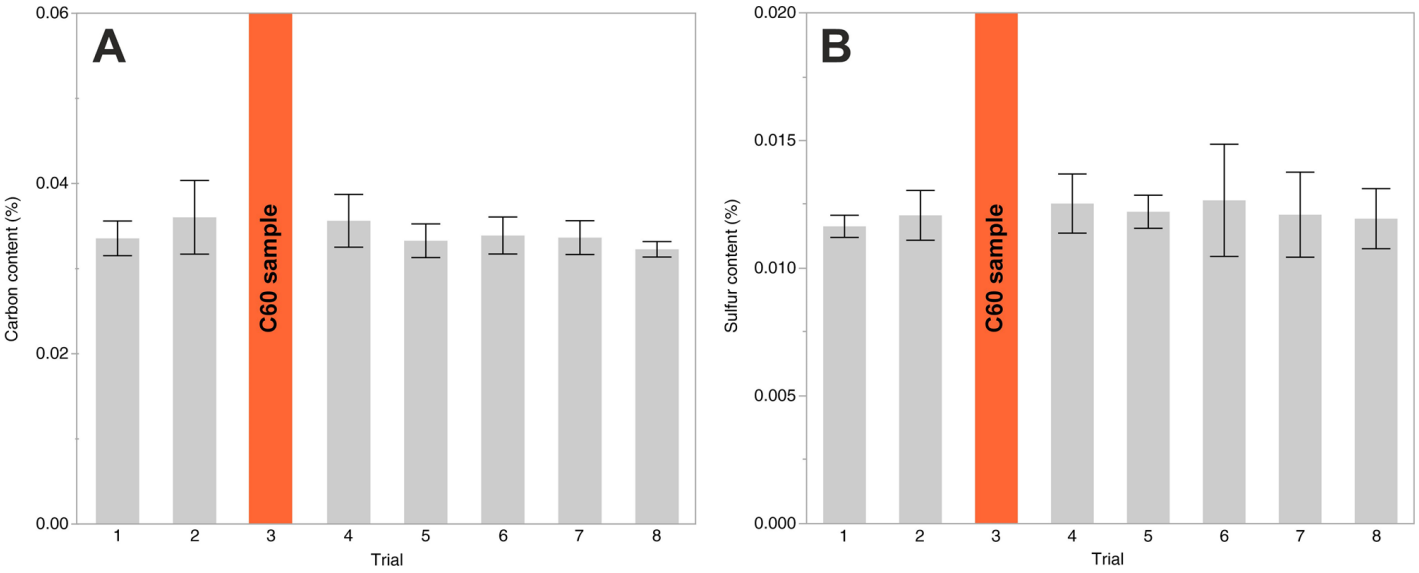

Figure 3: *(A) Grey bars show the mean percentage C content ± standard deviation of RH 31/32 samples, the red bar represents the insertion of the C60 sample (B) grey bars show the mean S content ± standard deviation.*

2. Impact of insertion of a RH31/32 sample into a series of C60 samples

The mean C content after preparation of C60 samples was between 0.5992 ± 0.0199 % and 0.6061 ± 0.0197 % (Table 2, Figure 4). Insertion of a RH31/32 sample caused a decrease of the C content to 0.0389 ± 0.0060 %. The only significant difference could be observed

between trial 3 and all other trials (t-test group B, Table 2). By contrast, trials after preparation of C60 sample were not significantly different (t-test group A).

The mean S content for trials with C60 samples was between 0.0266 ± 0.0005 % and 0.0273 ± 0.0003 %. Insertion of the RH31/32 sample resulted in a decrease of the S content to 0.0085 ± 0.0006 %. Only trial 3 (t-test group B, Table 2) was significantly different to trials using C60 samples (t-test group A).

Trial	Carbon			Sulfur		
	Mean ± SD	t-test group		Mean ± SD	t-test group	
1	0.6023 ± 0.0186	A		0.0266 ± 0.0005	A	
2	0.5992 ± 0.0199	A		0.0272 ± 0.0007	A	
3	0.0389 ± 0.0060		B	0.0085 ± 0.0006		B
4	0.6018 ± 0.0056	A		0.0273 ± 0.0003	A	
5	0.6002 ± 0.0141	A		0.0273 ± 0.0003	A	
6	0.6017 ± 0.0267	A		0.0269 ± 0.0009	A	
7	0.6061 ± 0.0197	A		0.0270 ± 0.0002	A	
8	0.6048 ± 0.0172	A		0.0269 ± 0.0008	A	

Table 2 (right): *Mean percentage content ± standard deviation for C and S in each of the eight trials of the second test series. Trials sharing the same test-group character showed no significant difference (t-test).*

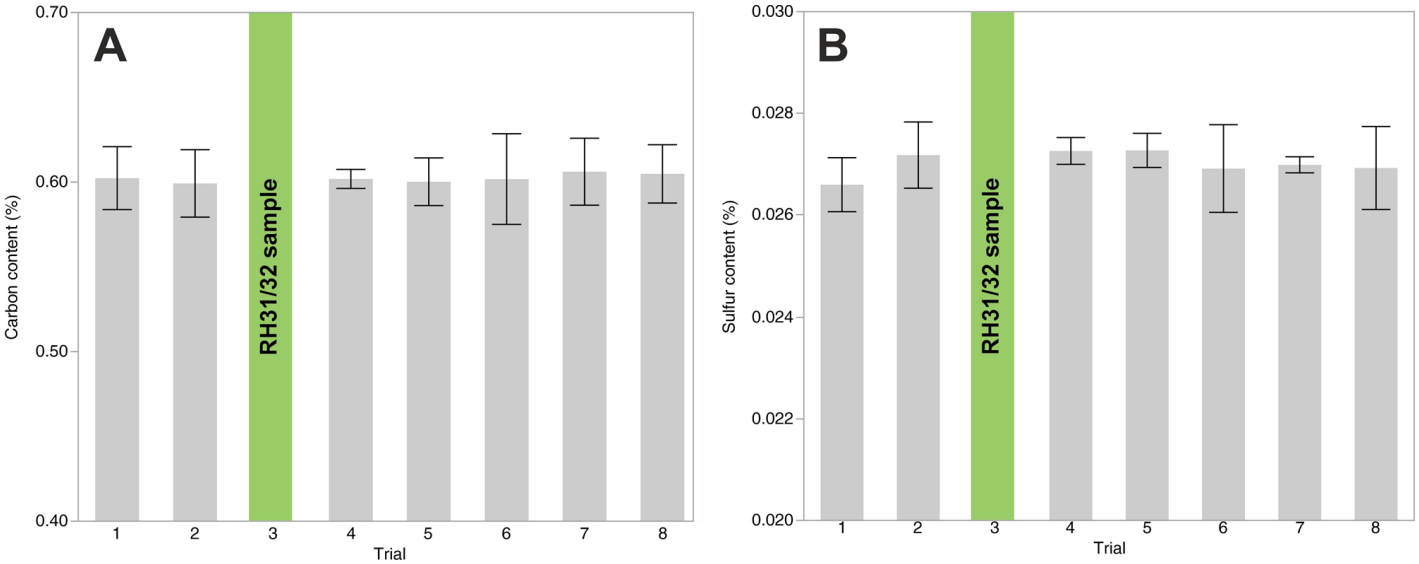

Figure 4: *(A) Grey bars show the mean percentage C content ± standard deviation of C60 samples, the green bar represents the insertion of the RH31/32 sample (B) grey bars show the mean S content ± standard deviation.*

Discussion

This study demonstrates that the automatic production, collection, transport and reception of chips did not cause any contamination as measured by thermal evolution analysis. This was verified by a series of trials on samples with low C and S content which was interrupted by sample with high C and S content. Contamination due to the sticking of chips from the previous sample with high C and S content should have led to an increase in the element concentration in subsequent samples. No such increase was detectable. The reverse case, introduction of a low-concentration sample into a test series with a high C and S content, also produced no evidence of contamination.

Internal investigations, which have not been presented here, show that contamination can be detected by combustion analysis from a percentage mass content of 0.3 %. This corresponds to approx. 1.5 mg material or about one to two granular chips. The results of our investigations indicate that contamination with even such small amounts can be prevented by using our technology. Visual inspection of the chip samples also did not reveal any indication of chips that could have originated from the previous sample.

The avoidance of contamination is based on a combination of two key factors. On the one hand, the smooth and short shape prevents the chips from getting caught on the inside of the transportation tube and other contact surfaces. On the other hand, all elements that may come into contact with chips are carefully designed to minimize deposition and sticking. Both factors together make it possible to use the technology in fully automatic mode.

The results presented here were obtained on samples with specific material properties. For each sample the material properties must be taken into account to choose the optimal method of sample preparation. For example, particularly soft material tends to stick to the cutting edge of the milling plates.

In this case, it may be necessary to discard the first chip batch of the subsequent sample in order to avoid contamination by chips stuck to the cutting edge. The software of the HS-F 1000 and the PrepMaster system offers all possibilities to configure and adjust the sample preparation to achieve contamination-free analytical results.

References

[1] HERZOG Application note 38/2021: A novel approach for thermal evolution analysis of steel samples by using chips with granular morphology.

[2] HERZOG Application note xx/2021: Combustion analysis of granular steel chips The effect of the scale layer on the analysis of carbon and sulfur

Germany

HERZOG Maschinenfabrik
GmbH & Co.KG
Auf dem Gehren 1
49086 Osnabrück
Germany
Phone +49 541 93320
info@herzog-maschinenfabrik.de
www.herzog-maschinenfabrik.de

USA

HERZOG Automation Corp.
16600 Sprague Road, Suite 400
Cleveland, Ohio 44130
USA
Phone +1 440 891 9777
info@herzogautomation.com
www.herzogautomation.com

Japan

HERZOG Japan Co., Ltd.
3-7, Komagome 2-chome
Toshima-ku
Tokio 170-0003
Japan
Phone +81 3 5907 1771
info@herzog.co.jp
www.herzog.co.jp

China

HERZOG (Shanghai) Automation Equipment Co., Ltd.
Section A2, 2/F, Building 6
No. 473, West Fute 1st Road,
Waigaoqiao F.T.Z., Shagnhai,
200131
P.R.China
Phone +86 21 50375915
info@herzog-automation.com.cn
www.herzog-automation.com.cn