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Automated Auto-Ignition Temperature Measurement with Optical Flame Detection

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Abstract: An automated auto-ignition installation in accordance with the safety standards described in DIN 51 794 and CENELEC EN 50014 CLC detecting the appearance of a flame and the controlling testing program using LabVIEW software package were developed and tested. The results obtained are reproducible. Its main advantage is time-saving.

Keywords: Auto-ignition · Automation · Optical flame detection

Introduction

Firmenich industries proposed, as a topic for diploma thesis, the measurement of auto-ignition temperatures and the appearance and detection of the flame by an automatic process. Indeed, both experimental observations are essential to comply with official certification of these measurements.

A first study for automated control of the temperature with the use of the software LabVIEW (Laboratory Virtual Instrument Engineering Workbench) had been done in 1994. However, the surveillance and the registration of the appearance of the flame were not yet available.

Need for Measurements

The auto-ignition temperature (AIT) of a substance is the temperature at which the vapour ignites spontaneously from the heat energy of the environment. The first measurements of auto-ignition were performed at the beginning of the 20th century to avoid explosions in the coal mines or when handling gaseous mixtures.

The concern for security and environmental implications has resulted in the creation of legal and standardised requirements for industry to know the physico-chemical properties of substances they handle and store. The AIT is one of the safety characteristics; unexpected and violent reactions are one of the causes of major accidents, like the explosion of the AZF factory in Toulouse (2001) where 30 people lost their lives and more than 1000 people were injured.

Measurement of the AIT is a time-consuming operation. Among other methods to determine the auto-ignition temperature, a normalised method is defined in DIN 51794. An Erlenmeyer flask is heated in an oven for five minutes and a known quantity of solvent is introduced. The temperature is measured in the container with a thermocouple (defined by the DIN norm) and when ignition occurs, the temperature changes drastically. The maximum delay for ignition is five minutes. If no ignition occurs, the temperature is increased stepwise.

Controlling the procedure with the software LabVIEW was a first step in automation. The given temperature was first increased to 40 °C, then when a reaction was registered, the temperature was fixed between the two last points and so on, following the sequence illustrated in Fig.1.

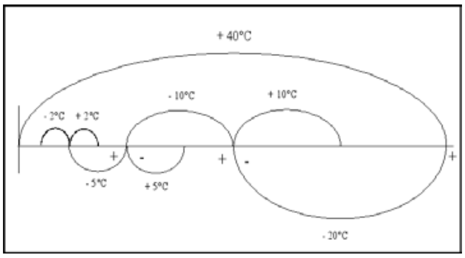

Fig. 1. Iteration routine used to determine the region of the AIT at a fixed volume

Flame Detection

To improve the automation, it was necessary to check not only the auto-ignition but also the presence of a flame, because a reaction can occur when the substance

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comes into contact with the container and this causes a temperature jump without ignition. The addition of an optical detector was investigated. The first idea was the installation of a webcam that could take pictures of the flame. However, some substances burn with an invisible flame or 'white flame', such as diethyl ether.

The original part of this work is the detection of the flame. Two optical detectors are installed, one covering the visible spectrum (350–820 nm), and a second one to detect in the ultraviolet domain (200–400 nm) for the 'cold flames'.

Automation

The automation undertakes the following tasks:

- registration of the product data given by the operator;
- regulation of the heating of the oven;
- control of the intake of samples and their dilution;
- detection of auto-ignition;
- production of a test report for each experiment.

The LabVIEW software is well suited for data acquisition and instrument control. Fig. 2 illustrates the front panel designed with LabVIEW to program different control parameters and follow the acquisition data.

Results

Different series of tests for different substances were first performed at a fixed injection volume and then at a range of volumes. The results were compared with those published in the literature.

As an example, the results obtained for n-heptane are given in the Table compared to literature values. The first value corresponds to the final recorded AIT; the second value corresponds to the time lag in seconds

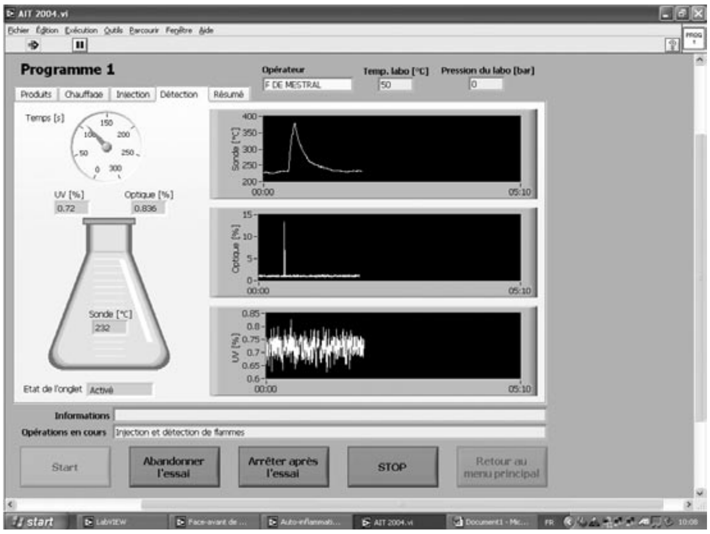

Fig. 2. Measurement of n-heptane: 221 °C ± 2.5 °C

and to the injected volume; the last value is the calculated activation energy after regression of all time lags recorded as a function of the measured AIT.

The tests were performed several times and the results shown were promising.

Conclusion

This research and development gives good results which are in agreement with the literature and those obtained at the Firmenich safety laboratory and the method corresponds to DIN 51794. Furthermore, the advantage of a fully automated evaluation leads to a significant gain in time required for analysis procedures as well as to the possibility of screening a large number of new substances of great interest to industry.

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Table.

Substance	**Merck**[a]	**Chapman & Hall**[b]	**SAX 9th Ed.**[c]	**HSDB**[d]	**Firmenich Safety Lab. DTDC**	**This work: Last AIT recorded with a fixed volume**	**This work: Last AIT with a variable volume**
n-Heptane, C_7H_{16} CAS 142-82-5 Fluka, >99% (GC)	215 °C	204 °C	223 °C	-	221 °C 95 s with 250 µl 114 kJ·mol^{-1}	221 °C 95 s with 250 µl 114 kJ·mol^{-1}	216 °C 140 s with 300 µl n.a.

[a]CD-ROM: Merck Safety data sheet Database, Version 1, 1996
[b]CD-ROM: Chapman & Hall, Dictionary of Organic Compounds, Version 5:2, 1997
[c]CD-ROM: SAX's Dangerous Properties of Industrial Materials, Ninth Edition, 1995
[d]CD-ROM: U.S. National Library of Medicine, CHEM-BANK - Databanks of potentially hazardous chemicals, November 1994