Detection of ammonia leak in laboratory conditions using a FLIR 306 camera and a portable gas detector

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Abstract
The study aimed to analyse detection methods of ammonia leakage by various electrical methods. Rapid detection of the escape of hazardous technical gases is extremely important in large chemical plants. The basis of the research was the use of a specialized camera, model FLIR GF306, to detect a leak of selected gases in a narrow infrared band. In laboratory conditions, the controlled emission of gaseous ammonia at various concentrations was simulated and gas detection was performed: a) using a narrow infrared thermography method, b) by a portable electrochemical detector dedicated to detecting ammonia. The turbulent flow of gas into the environment and high thermal contrast between the expanding gas and the background are the conditions for effective gas detection with a thermal imaging camera operating in a narrow infrared band.

Keywords: Thermal imaging, infrared, gas detection, ammonia

Introduction

In large chemical plants, ammonia gas may leak from equipment and transmission pipelines. Rapid detection is mainly carried out by using electrochemical detectors. They are mounted stationary and cooperate with the monitoring system. The electrochemical detectors are also used as portable devices for monitoring the condition of installations and controlling employees' safety. Ammonia is toxic and harmful to employees and the environment, and its emissions must be minimized. A new technique that can improve safety at chemical plants is the detection of gases, including ammonia, based on thermography in a narrow spectrum band [1, 2]. Monitoring of installations and gas emissions can be carried out by using portable thermal imaging cameras designed for the rapid detection of working gas leakage. Thermal imaging cameras are used in medicine, by the army, the police and customs officers (detection of people, objects, substances) [4, 5], in the industry (for process assessment, failure detection) [6, 7], in power engineering (for diagnostics of device status and detection of emergency conditions) [5, 8, 9]. Cameras for thermal imaging are most often equipped with the Focal Plane Array detector type [2, 5, 6]. In modern cameras, matrixes up to 1024 × 768 pixels and image refresh rates from 0.0015 Hz are used [14, 15]. Such a large number of obtained thermograms allow for the registration of ultra-fast thermal processes such as the detection of gas leaks. Detectors in the matrices have different properties. The optical sensitivity of the detector depends on its chemical composition. Describing the behavior of gas, the pressure depends on the temperature and the volume, and also the volume depends on the temperature and the pressure. The internal energy (for the black body) depends on the temperature and volume if those are the variables, or it depends on the temperature and the pressure, or the pressure and the volume [3]. Due to the Clausius-Clapeyron equation, it can be calculated how much energy will flow per second from the gas container (where the gas is under pressure) through the hole of the unit area. The equation for total energy density (the flux from a small hole per unit area) is:

$$\frac{U}{V} = \frac{4\sigma}{c} T^4 \tag{1}$$

where:
U is the total energy of all the photons;
V is the volume of the gas container;
σ is the Stefan-Boltzmann constant:

$$\boldsymbol{\sigma} = \frac{k^4 \boldsymbol{\pi}^2}{60\hbar^3 c^2} = 5.67 * 10^{-8} [\frac{W(m)^2}{K^4}]$$

c – the speed of light, constant: c = 299 792 458 [$\frac{m}{s}$];
T – temperature [K]
[3].

When the gas flows through a small hole into the environment, it expands intensively, and the temperature decreases. The largest pressure fluctuations, and thus temperatures, occur during turbulent gas flow [3].

The FLIR GF306 optical gas imaging camera (Fig.1.) is capable of detecting anhydrous ammonia (NH_3). It visualizes gas emissions without the need to shut down operations. The portable, non-contact IR camera GF306 allows scanning installation for leaks while maintaining a safe distance from the

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equipment. By catching leaks early, the GF306 reduces lost revenue to breakdowns and repairs. GF306 inspections are faster than traditional leak-detection tools. The camera can work in several modes. Thermal images are recorded as a) individual thermograms, b) high-sensitivity mode (generation of differential images), c) thermal film recording mode (in MP4 format). In differential, high-sensitivity mode, the camera records, and processes thermogram sequences. It is not the thermogram that is displayed but the image resulting from mathematical operations (i.e. the differential image). In this mode, the next frame (or several frames) is "subtracted" from the previous thermogram in the sequence, and then only the points that remain from this operation are displayed and they form the image. The registration of thermal films (with a refresh rate of approximately 60 Hz) makes it easier for the observer to notice the movement of the object which is the movement of gas fumes against the background. Thus, an important element of correct measurements is to set the camera in a stationary position and observe (or record) the gas movement against a uniform background. The camera has no alarm function. The manufacturer does not provide recommendations regarding the measurement environment (whether it is possible to carry out measurements in closed or open spaces), and what the impact of cooling or heating on the detection efficiency is. The cost of buying the FLIR GF306 camera with additional equipment and software is very high (it is about 100,000 Euro in 2019; to compare, the price of a single-gas ammonia detector is about 250 Euro). On the other hand, companies can rent a camera, for a single checkup, which significantly speeds up inspections and maintenance of installations, especially in large plants. The high cost of gas detection cameras as well as poor knowledge of their applications are the biggest obstacles to the dissemination of this technique [6, 7].

Materials and Methods

As part of the study, ammonia gas was emitted at various concentrations under controlled conditions. The environmental conditions in the laboratory were as follows: temperature from 6 to 20°C, and 50% humidity. The measurements were carried out at various distances from 0.5 m to 2 m. The distance was adjusted so that the recorded images represented the detected substance in the best possible way. A series of detections of the tested gases with the FLIR GF306 camera was performed. Images were recorded in three camera modes: a) individual thermograms, b) series of differential images, c) high - resolution thermographic films. The research was conducted in cooperation with Grupa Azoty S.A. in Tarnów and the EC Test System company. In the first part of the tests, the FLIR GF306 camera was tested to detect ammonia in the air under conditions of gas emissions of known concentration. Thermograms of gas vapors were recorded in a room, under a constant temperature fume cupboard (around 20°C) as well as against the background of thermal contrast which was an open window (outside temperature was around 6°C). About 40 thermograms, 2 film recordings in MP4 format, and 2 thermal image sequences were registered. The HovaCAL calibration gas generator was used to adjust the concentration value, shown in Figure 2. The gas generator was controlled by the ViewCal 411 software [10]. During measurements of ammonia at lower concentrations, a control measurement with a single-gas detector by GasAlert Extreme Ammonia Meter by BW Technologies was additionally performed, which is shown in Figure 3 [11]. This detector operates relying on the use of electrochemical sensors, and detects the presence of ammonia in the range of 20-100 ppm. Ammonia was taken from gas cylinders (with Air Products certificate, the content of ammonia in the cylinder was 7% by volume (in N_2). The detection test was carried out with the generation of dry ammonia at the concentration of 300 ppm, 946 ppm, 3000 ppm, and 7000 ppm in turn. The gas flow was laminar and varied from 4 liters per minute to 10 liters per minute.

Figure 1. Thermal camera FLIR GF306; [6]

Specification of the FLIR GF306 camera used for measurements is presented in table 1. The equipment is applicable to detect about twenty selected gases and organic compounds, such as NH_3 or SF_6 [6].

Figure 2. HovaCal – calibration gas generator; [10]

Table 1. Parameters of the FLIR GF306 camera, for detection of selected gases and organic compounds; [6]

Detector type	Focal plane array, QWIP (cooled)
Spectral range	10.3 – 10.7 µm
Resolution in Infra Red	320 x 240 pixels
Thermal sensitivity	< 15 mK (at 30°C)
Detector/ Frame range	60 Hz
accuracy	±1°C *(±1.8°F)* for temperature range (0°C to 100°C) or ±2% of reading for temperature range (>100°C)
Minimum Laboratory Leak Rate (MLLR) for known gases	Ammonia: 0.127 g/h
Temperature range	-40°C to + 500°C
Dynamic Range	14-bit, Real-time non-radiometric recording: MPEG4/H.264
Automatic Gain Control	Continuous/manual, linear, histogram
Lenses (FOV) / focuse optimum	Standard: 24° (23 mm); 0.3 m
zoom	1-8× continuous digital zoom
focus	Automatic (one touch) or manual (electric or on the lens)
Radiometric JPEG	14-bit measurement data included
Radiometric IR Video	7.5 and 15 Hz . seq video clips to memory card
Environment temperature range	-20°C to + 20°C

Figure 3. GasAllert Extreme Ammonia Meter - detector; [11]

The thermographic camera manufacturer's software, FLIR Tools were used to analyse the thermograms. The thermal image in differential mode shows the points where the corresponding gradient of temperature changes over time was recorded. The black and white scale corresponds to points with a high-temperature gradient (black), and a lower temperature gradient (white).

Results and Discussion

The first attempt to detect ammonia with the FLIR GF306 camera was made for dry gas at a concentration of 300 ppm and gas flow at a rate of 4 liters per minute. This corresponds to an NH_3 ammonia flow of 50 g/h, i.e. well above the detection threshold indicated by the manufacturer: 0.127 g/h. The gas flowed gently from the evaporator tube, propagating in the space under the fume cupboard. On the recorded individual thermograms (Fig. 4.), as well as in thermal films, or in the high sensitivity mode (HSM, i.e. the mode of differential thermogram generation) as shown in Fig. 5. There was no flow of "smoke" that would testify the presence of gas. Therefore, it can be concluded that the FLIR GF306 camera did not detect ammonia emissions in the conditions described, for a concentration of 300 ppm. For the simultaneous additional control of the concentration of ammonia in the air, a single gas GasAlert detector was used to detect ammonia. The detector was placed at a distance of about 20 cm from the leakage site, i.e. in our case from the end of the tube led out from the HovaCal calibration gas generator. The GasAlert single gas detector already detected ammonia at 20 ppm. This concentration of gas was also felt by the sense of smell of a man.

In the next test, no gas was detected with the FLIR GF306 camera at a concentration of 946 ppm and laminar gas flow from the evaporator under a constant temperature of 20^{o}C under the fume cupboard. The escaping gas quickly mixed with air and its concentration decreased. The next gas detection test was made in a laboratory, in which the temperature was 20^{o}C close to an open window, with the temperature outside about 6^{o}C. The sky was cloudy, which gave a uniform background for the detection. For measurements at a flow of about 4 liters per minute and a dry ammonia concentration of 300 ppm and 946 ppm, the detection was also difficult. The gas flow for subsequent measurements was increased to 10 liters per minute. The first leaks could be observed at the concentration of 3000 ppm. Despite such a high concentration, the leak was practically invisible in individual thermograms, which can be seen in Figure 6.

Figure 7 shows the image obtained from the FLIR GF 306 camera in the high-sensitive mode (HSM), i.e. the mode of generating and displaying differential images in the camera software. The camera in this mode records and processes thermogram sequences. It is not the thermogram that is displayed but the image resulting from mathematical operations (i.e. the

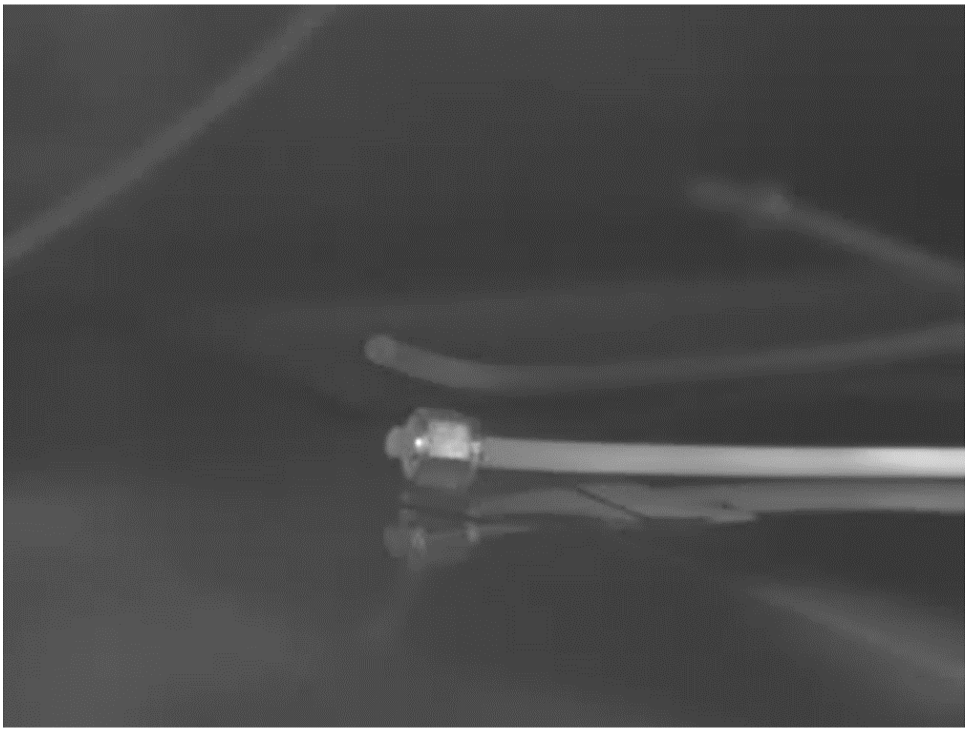

Figure 4. Thermogram of laminar emissions of ammonia at the concentration of 300 ppm, at the constant ambient temperature of 20°C. FLIR GF306 camera

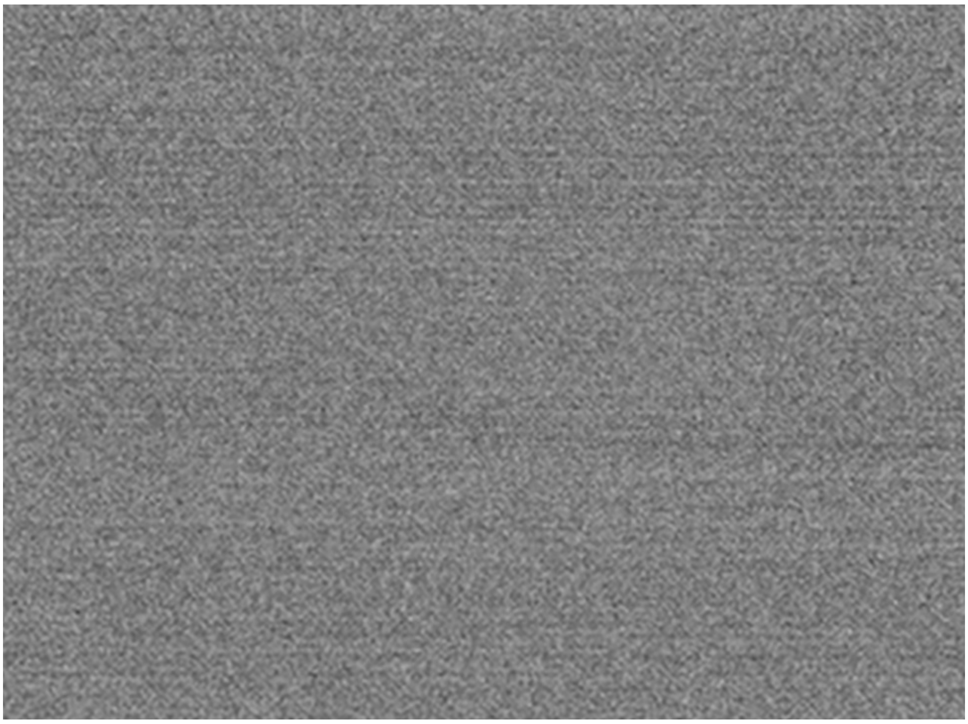

Figure 5. Highly sensitive differential mode (HSM) thermogram of laminar emissions of ammonia at 300 ppm at the constant ambient temperature of 20°C. FLIR GF306 camera

differential image). The use of differential mode in the image camera software improves leak visualization.

Noticing differences in the image, even in differential image mode, is quite difficult for such a value since the differences are very subtle. However, this is possible with the rapid emission of subsequent differential thermograms in the camera. In the next step, the gas concentration at the nozzle outlet was increased to 7000 ppm. By increasing the gas concentration and using the differential mode, gas leaks in the form of swirling smoke can be clearly observed, as shown in Figures 8 -10. This gas leak can be seen in thermographic films recorded during the test: **https://youtu.be/TkmWRErr9hs** .

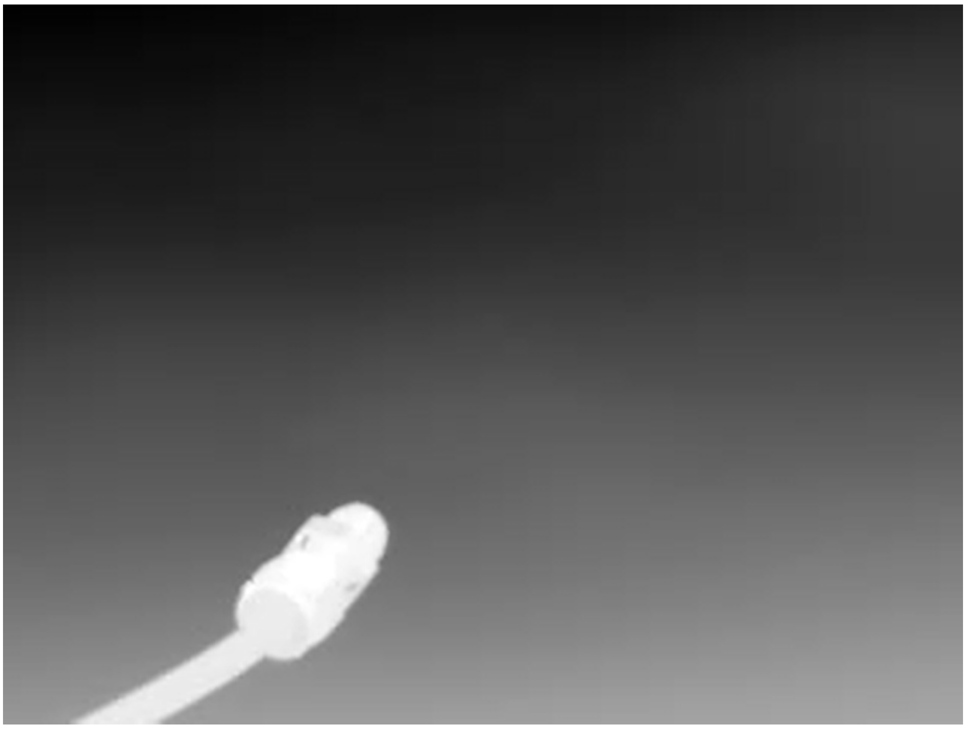

Figure 6. Thermogram of **laminar** emission of ammonia at the concentration of 3000 ppm, at the ambient temperature of 20°C in the **background area at 6°C**. FLIR GF306 camera

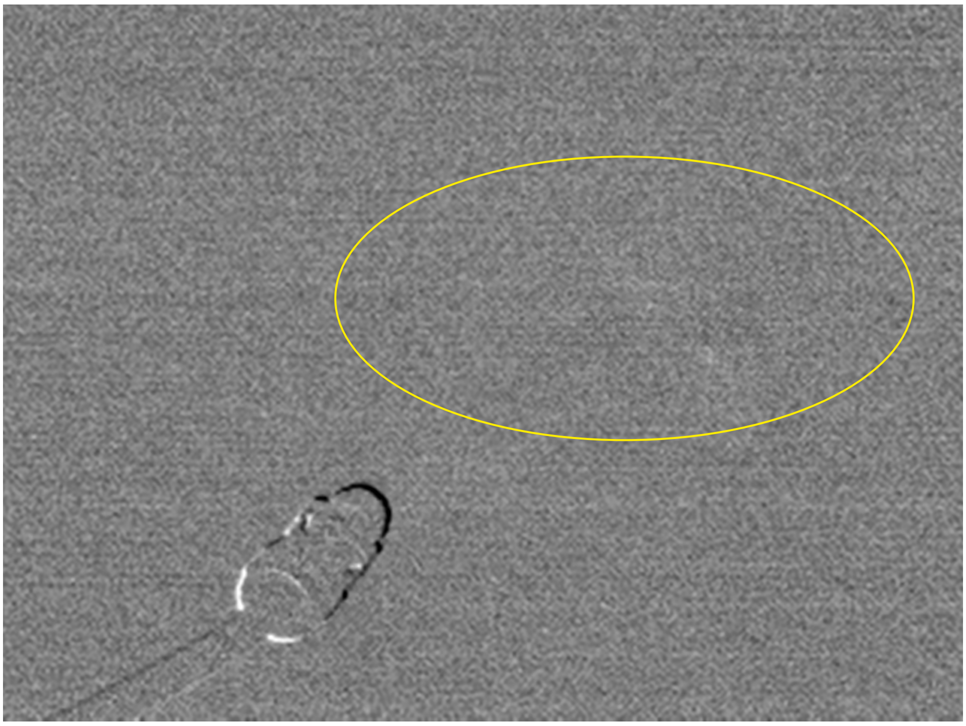

Figure 7. Thermogram of **laminar** emission of ammonia at the concentration of 3000 ppm, at the ambient temperature of 20 °C in the **background area at 6°C**. FLIR GF306 camera

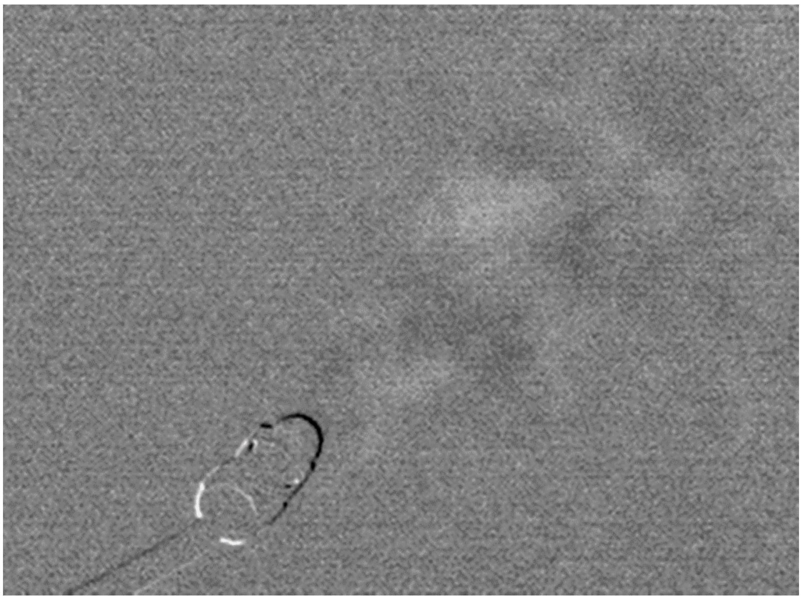

Figure 8. Highly sensitive differential mode (HSM) thermogram of **turbulent** emissions of ammonia at 7000 ppm at the ambient temperature of 20°C, against the **background area at 6°C**. FLIR GF306 camera

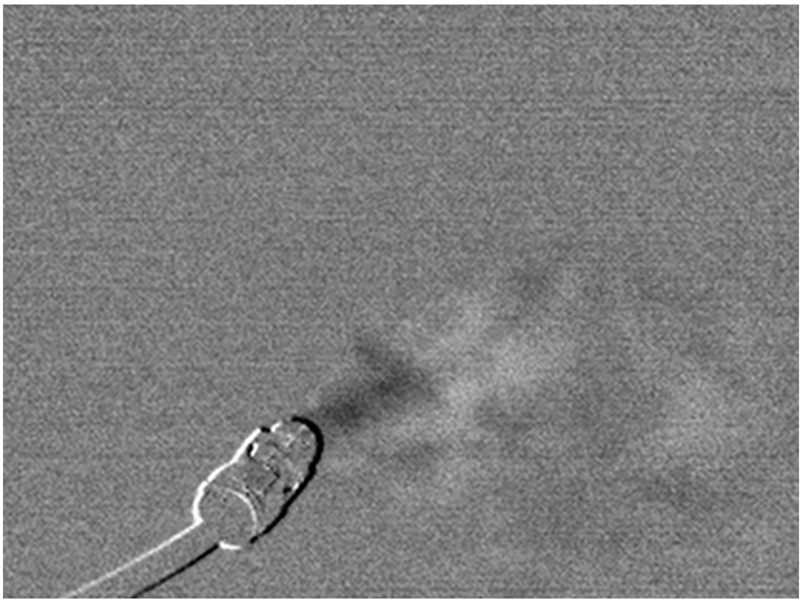

Figure 9. Highly sensitive differential mode (HSM) thermogram of **turbulent** emissions of ammonia at 7000 ppm at the ambient temperature of 20°C, against the **background area at 6°C**. FLIR GF306 camera

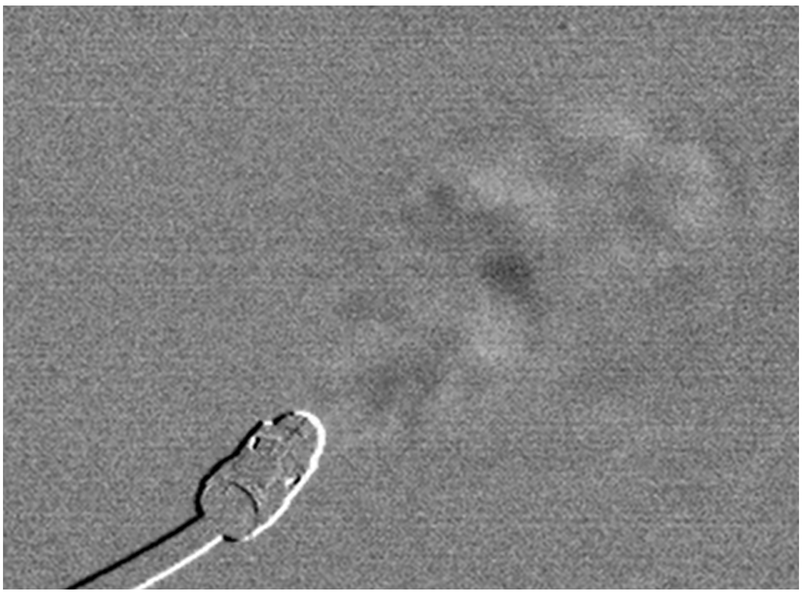

Figure 10. Highly sensitive differential mode (HSM) thermogram of **turbulent** emissions of ammonia at 7000 ppm at the ambient temperature of 20°C, against the **background area at 6°C**. FLIR GF306 camera

Conclusions

– The FLIR GF306 gas detection camera has successfully detected ammonia in the range 3000–7000 ppm.
– The condition for effective gas detection is the turbulent leak of gas into the environment.
– The condition for effective detection is a high thermal contrast between the expanding gas and the background.

Author Contributions

Conceptualization, methodology, validation, formal analysis, writing – original draft preparation, data curation, writing – review and editing, supervision, project administration, funding: Agnieszka Lisowska-Lis. Investigation, resources, acquisition, visualization: Agnieszka Lisowska-Lis and Piotr Siudut.

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