

# POSSIBILITIES OF USING TRACER GAS

## MOŽNOSTI POUŽITÍ ZNAČKOVACÍHO PLYNU

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### Abstract

The paper describes hexafluoride sulfur ( $SF_6$ ) as a chemical compound and its chemical attributes. It also describes in detail a way of using  $SF_6$  as a tracer gas for analyses of mining ventilation.  $SF_6$  was investigated by the Bureau of Mines in the USA together with other organic and inorganic compounds and radioactive substances.  $SF_6$  was found as a suitable tracer gas. The next chapter deals with the application of  $SF_6$  (in situ) at the Frenštát Mine.

### Abstrakt

Tento článek ve stručnosti popisuje hexafluorid síry ( $SF_6$ ) jako chemickou sloučeninu a jeho chemické vlastnosti. Podrobněji je v tomto příspěvku popsán způsob využití  $SF_6$  jako značkovací plyn při analýzách důlního větrání.  $SF_6$  byl zkoumán Báňským úřadem v USA spolu s ostatními organickými i anorganickými sloučeninami a radioaktivními látkami.  $SF_6$  byl shledán vhodný jako značkovací plyn. Další kapitola pojednává o vlastním použití  $SF_6$  (in situ) na Dole Frenštát.

**Key words:** Mine Ventilation, Tracer Gas, Return Airway, Mine Air Recirculation.

## 1 INTRODUCTION

The United States Bureau of Mines utilizes  $SF_6$  as a tracer gas to measure mine atmosphere under conditions, where conventional methods have failed.  $SF_6$  has been experimentally used for measuring the recirculation of intake and return air induced by escape through old workings, determining a potential release from adjacent mines, monitoring air losses from the intake airway and measuring the time of air infiltration through uranium mines. It is an applicable aid for accurate measurements of volume flow rate of air through airways of large profiles at very low speeds air circulation and associated with underground cooling devices.

## 2 $SF_6$ SULFUR HEXAFLUORIDE

$SF_6$  is a compound of sulfur and fluorine, and is thus a synthetic substance. This gas is colorless and odorless. Under normal conditions it is stable.  $SF_6$  is non-inflammable, non-toxic and non-corrosive. Under pressure it becomes liquid. In contact with a liquid it can cause skin damage. High concentrations may cause suffocation. Symptoms are then locomotor disorders and loss of consciousness. When it burns, thermal decomposition may produce toxic or corrosive substances, such as hydrogen fluoride or sulfur dioxide.

Its molar mass is 146 g/mol. Its pour point is  $-50.8$  °C, boiling point is  $-63.8$  °C, critical temperature is  $45.5$  °C, solubility in water 41 mg/l. Its vapors are nearly five times heavier than air. In confined space they can gather especially on the floor or in low-lying areas.

## 2.1 Use of the tracer gas in analyses of mine ventilation

Using  $SF_6$  as a tracer gas can be an effective means of solving the above mentioned ventilation issues. The United States Bureau of Mines deals with searching these issues and their solutions, including air programs. For the purpose of applicability of the tracer gas  $SF_6$  various studies have been carried out at several mines.

For detection purposes both organic and inorganic compounds and radioactive substances have been used, however many of these substances showed fundamental deficiencies. Chemical tracers are less detectable than the concentration of radioactive substances, and are often highly absorbed by many surfaces. On the contrary, radioactive substances can be detected in low concentrations, but they can be treated worse and are not suitable for work underground. Effective tracer gas must be detectable in low concentrations, must be safe, have a low concentration background and must be chemically and thermally stable.

These basic requirements are fulfilled by sulfur hexafluoride. It can be detected at very low levels using the chromatograph with electron capture detection. Saltzman [1] Niemeyer and McCormic [2] have shown that  $SF_6$  can be detectable already at a concentration of  $10^{-5}$  ppm. Lester a Greenberg [3] have shown that  $SF_6$  is safe for the survival of rats at 80% of  $SF_6$  in the atmosphere for 24 hours and even disease-free.

Hunt and Moore [4] have shown that  $SF_6$  is not measurably absorbed by sandstone deposits. Whisman [5] has presented the same for coal deposits. Finally,  $SF_6$  has the indisputable advantage that it does not occur in a natural atmosphere.

## 3 RELEASE AND MEASUREMENT OF $SF_6$

Also the process of  $SF_6$  release into the mine atmosphere has been studied. The main problem was in incomplete mixing of dense  $SF_6$  with the mine atmosphere in the airway with a low volume air flow rate. At higher speeds of air circulation this mixing problem did not occur.

Good miscibility of  $SF_6$  with the mine atmosphere is achieved, when  $SF_6$  is released in the form of flow from a pressure container through a small hole (0.0024 cm) drilled in the cap. The mixture is so better adapted to the transfer of the pressure container through the mine airway during discharge of  $SF_6$ . The  $SF_6$  volume released is determined from the weight loss of the pressure container.



**Fig. 1** Pressure bottle with  $SF_6$  and syringes for samples

General calculation of  $SF_6$  released

The volume of sulfur hexafluoride is given by the weight loss of the container contents. The volume of the gas released is determined by the equation, as follows:

$$V = n.R$$

$V$  ... volume of  $SF_6$  released

$n$  ... mols of  $SF_6$  released

$R$  ... gas constant of  $SF_6$

This is a simplified version of the equation used previously:

$$PV = n.R.T$$

If  $n = \Delta M / M$

Where:  $\Delta M$  = bottle weight loss (g)

$M$  = molecular weight of  $SF_6$

Then the equation will be expressed, as follows:

$$V = \Delta M R / M$$

And if  $R=22,4$  l/mol

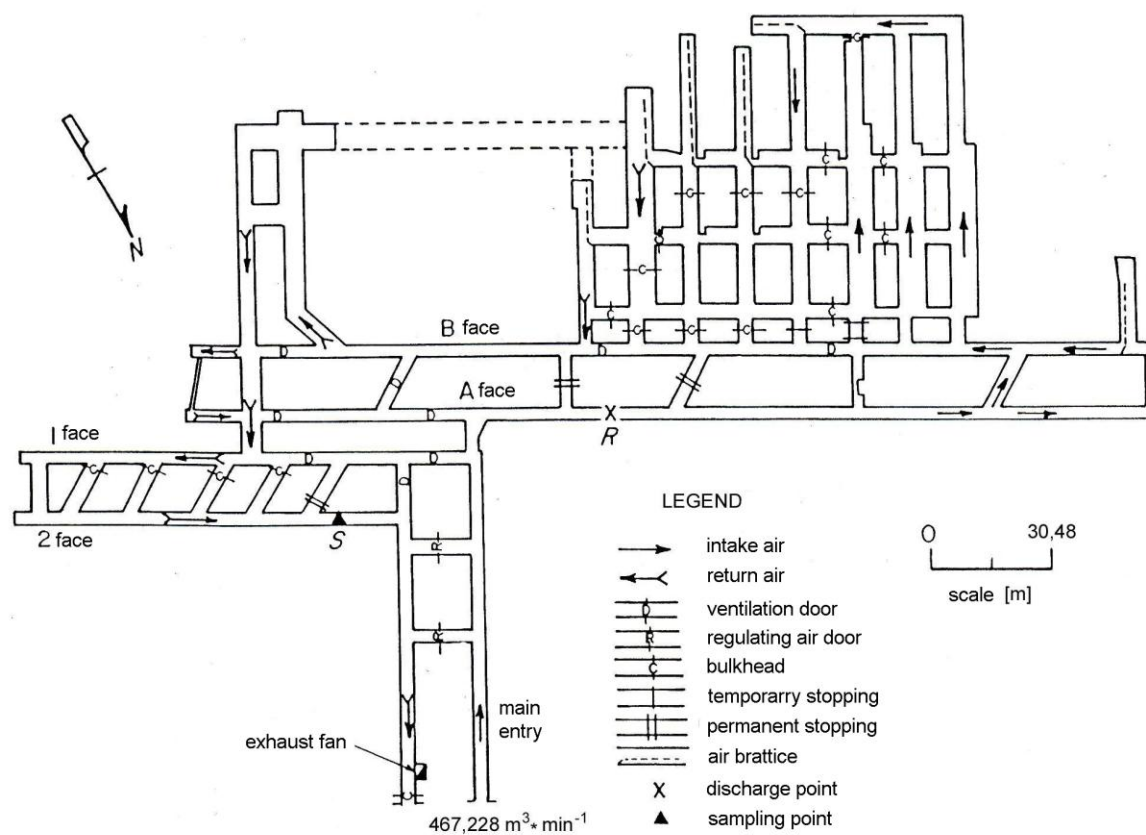
$M = 146,07$  g/mol

The equation will be reduced to  $V = 0,15\Delta M$

The determination of the released volume of  $SF_6$  is important for comparison of the quantity measured with the quantity released.

#### 4 PRELIMINARY EXPERIMENTS WITH THE TRACER GAS

In order to verify, whether  $SF_6$  can be determined quantitatively underground, the Bureau of Mines in Bruceton carried out preliminary experiments. The mine (Figure 2) has one intake shaft and one upcast hole and its place is ideal for testing the tracer gas. In this test, a known quantity of  $SF_6$  had been discharged into the intake air at point R and after several minutes samples were being taken from the return air at 2-minute intervals.



**Fig. 2** Experimental mine

The samples were tested using the chromatograph and the concentration of  $SF_6$  was determined. The quantity of  $SF_6$  passed in the return air was calculated by substituting appropriate values into the following equation:

$$Q_{SF_6} = Q_{vz} \int c dt$$

Where  $Q_{SF_6}$  is a volume of  $SF_6$ ,  $Q_{vz}$  is a volume flow rate of masses of air at the measurement point,  $c$  is a concentration of  $SF_6$  in time  $t$ .

The total time  $T$  is an interval of sampling (in this case 2 minutes) multiplied by the number of samples with a measured concentration of  $SF_6$ , then the equation above will be like this:

$$Q_{SF_6} = Q_{vz} \cdot c_{pnir} \cdot T$$

The experiment results were as follows: 10.5 l of  $SF_6$  was released into the intake shaft. 31 samples of 45 samples taken in the return airway at 2-minute intervals contained the measurable quantity of  $SF_6$  with the average concentration of 377 ppb. 31 samples containing  $SF_6$  indicated the presence of  $SF_6$  in the return airway for the period of 62 minutes at an average concentration of 377 ppb. The concentration of  $SF_6$  in the return airway is plotted as a function of time since the release of  $SF_6$  in the intake airway (Figure 3). The volume flow rate of the return air was  $0.4610 \text{ m}^3$  per minute. Using the continuity equation the calculated quantity of  $SF_6$  in the return airway is as follows:  $SF_6$  (litres) =  $(0.4610 \text{ m}^3 \text{ of air/min}) (9,5428 \cdot 10^{-9} \text{ m}^3 \text{ } SF_6 / \text{m}^3 \text{ of air}) (62 \text{ min}) (28,3 \text{ litres/ft}^3) = 10.7 \text{ litrů}$ . This is a good experimental match with the original value of 10.5 litres discharged into the intake airway. The result with such degree of accuracy was obtainable only when it was thoroughly mixed with the mine air.

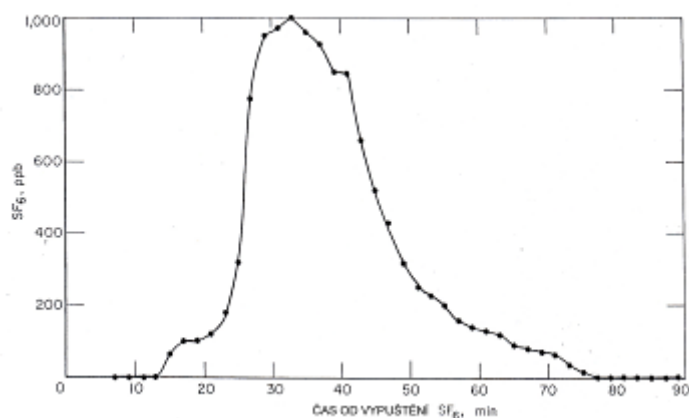


Fig. 3 Concentration of  $SF_6$  in the return air depending on time

## 5 MEASUREMENT OF AIR RECIRCULATION USING $SF_6$

At multi-level mines a serious problem may be the recirculation of the return air into the intake air in airways. In general, due to an escape through a mined-out area they are then randomly sealed. In case of a mine fire the air recirculation may then contaminate the intake air by toxic gases. To avoid this recirculation, we must first determine the extent to which it occurs, and then find the point of escape. For this purpose the tracer gas can be used.

Two gas experiments were carried out in the western ore mine, where the recirculation had occurred.

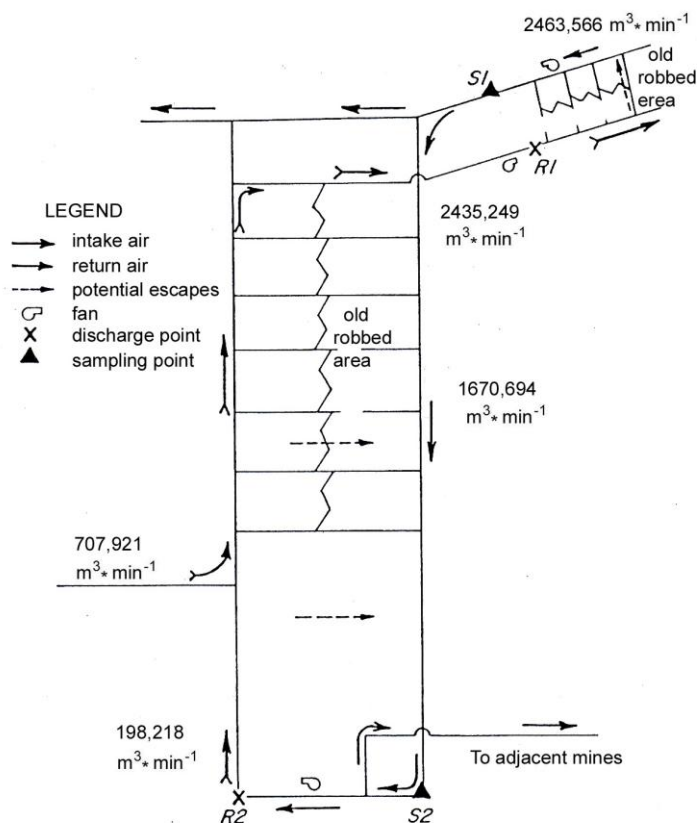
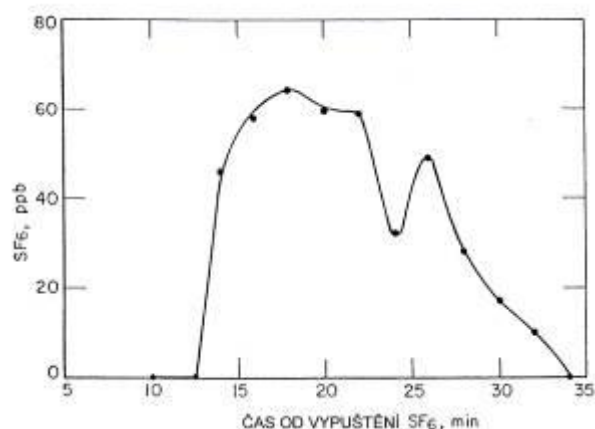


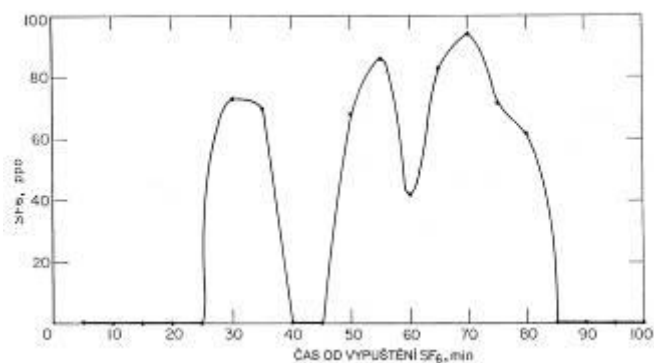
Fig. 4 Experimental mine

In the first experiment 34.2 l of  $SF_6$  was discharged in the period from 11.00 at point R1 into the return airway, where the air flow rate was  $2435 \text{ m}^3/\text{min}$ . Analysis of the sample showed that  $SF_6$  appeared first at point S1 at 10.59 o'clock and then in measurable concentrations up to 11.19 (Fig. 4). The calculated volume of  $SF_6$  passing through S1 was 2.1 l and confirmed the recirculation in this space, which is slightly higher than 6%.

In the next experiment of determining the recirculation, 8.6 l of  $SF_6$  was discharged, namely in the period from 13.10 to 13.23 at point R2 into the return air. The air flow rate at point R2 was  $198 \text{ m}^3/\text{min}$ , but then raised by the return air. Figure 5 shows the dependence of the  $SF_6$  concentration at point S2 on the time of release. The calculated volume of recirculated  $SF_6$  was 5.3 l, that means 61% recirculation.



**Fig. 5** Concentration of  $SF_6$  in the intake air during the first test of recirculation



**Fig. 6** Concentration of  $SF_6$  in the intake air during the second test of recirculation

### 5.1 Use of the tracer gas for measuring the air flow rate

One important application of the tracer gas serves to measure the volume air flow rate in airways, where for the standard measurement methods the diameter is too large, or the speed is too low. An experiment of this type was performed in Pennsylvania at a limestone mine with a vertical natural draft through an intake shaft about 21.33 m deep and 6.1 m in diameter. The shaft feeds the intake air from the surface down to an airway with a cross-section of  $74.32 \text{ m}^2$ . For measuring the air speed with an anemometer the cross-section was too large and the speed too low. Therefore, the measurement method was used by means of the tracer gas.

## 6 APPLICATION OF SULFUR HEXAFLUORIDE $SF_6$ AT THE LOCALITY OF THE FRENŠTÁT MINE.

At the Frenštát Mine locality sulfur hexafluoride -  $SF_6$  was applied to the mine areas. The quantity of discharged gas  $SF_6$  was determined according to the length of mine working behind the brattice stopping H6 at the discharge point, which is in total 65 m (31.6 m straight, 34 m turn left at an angle of  $120^\circ$ ). In this case, it was 24 litres of gas  $SF_6$ . The place of  $SF_6$  discharge was determined on the 3<sup>rd</sup> level of the shaft No. 5 at a depth of 1,042 m from the bank on the southern side behind the narrow permanent stopping No. H6, see Figure 7.

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*Paul F. ...*  
**Hráz čís. 6**  
(připravné - uzavírací)

*a říčky F5*  
Umístění hráze *32-570 m* sloj *—* vzdálenost od kříže *5 m* patro *3*

Popis provedení stavby hráze *říčka Mlýnská; stavba náhradní vodní dílny s přelivem, žebra ze stříbrné - železné, kování z ocel. prutu, upevnění betonem*

Rozměry záseků: hloubka *bez záseku na beton* šířka *—* hráz uzavřena dne *—*

Rozměry hráze i se zapuštěním: tloušťka *0,25* m, výška *9,0* m, šířka *7,0* m

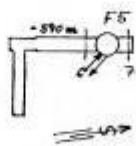
Rozměry otvoru: tloušťka *0,25* m, výška *1,2* m, šířka *0,8* m

Potřebná zásoba materiálu: *okovy a upevňovací prvky umístěny na pozemku*

Hráz postavena dne: *—*

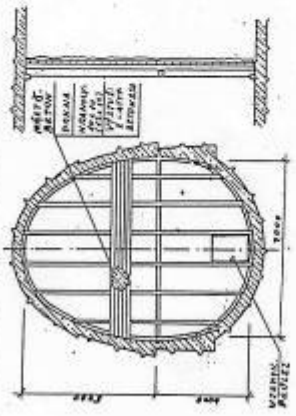
Hráz zrušena dne: *—* Způsob zrušení: *—*

Umístění hráze v dole - náčrty mapy v měřítku 1:2000 do vzdálenosti asi 50 m na všechny strany od hráze s mapovou sítí a označením směru větrného proudu:



*Od 15. 11. 1994 je říčka: F5*

Náčrty hráze v měřítku:

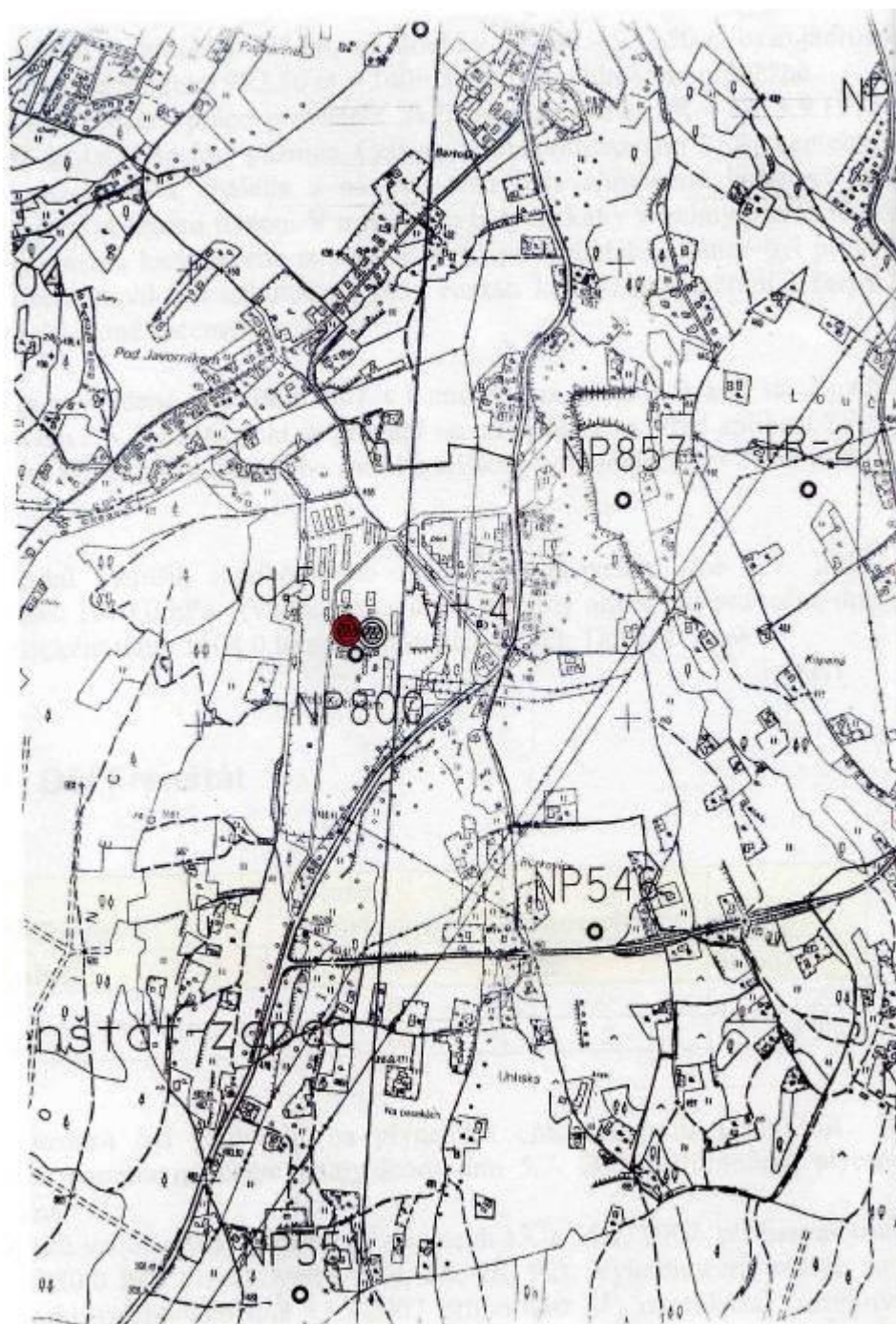


OKR - 11.3.16 MTZ D 21 88 R 801004

Fig. 7 Stopping No. 6



Subsequently, samples of air mass were taken within specified intervals and at a determined place. The sampling site was designed around the borehole No. NP 546, Trojanovice locality, see Figure 8.



**Fig. 8** NP 546 Trojanovice locality



The deep drill site is located in the cadastral area Trojanovice, 600 m southeast of the road Rožnov pod Radhoštěm – Frenštát pod Radhoštěm. According to a map documentation available at the map scale 1: 400 the crow-fly distance of the borehole from the shaft No.5 of the Frenštát Mine is about 920 m.

Drilling works were performed on:

- 14.5. 1973 – 25.7. 1973 by the T-50 drilling rig over the cover
- 7.5. 1974 – 8.7.1974 by the CR-1800 rig into carbon

Full-hole drilling was performed to a depth of 797.00, at a depth of 797.00 - 932.50 m the full-hole drilling was performed at intervals of 25 m. At a depth of 932.50 m - 1,409.50 m the full-hole drilling was performed continuously.

Pumping tests and liquidation work were performed by the FR-4 liquidation set in the period from 3.9.1975 to 25.2.1976. All casing pipes have remained in the borehole. A total of 32.8 t of PC 400 cement was consumed. The upper part of the casing pipes was burned and at the mouth of the borehole a concrete slab 120x120x30 cm was made 1 m below ground level. At the borehole site all necessary data was obtained on thickness and coal capacity of the Karviná formation. The borehole was even technically well made, so the full range of well logging and pumping tests could be accomplished there. The borehole was all properly cemented.

The application of  $SF_6$  was conducted on 29. 6. 2007 during the 1<sup>st</sup> shift on the 3<sup>rd</sup> level behind the tight H6 permanent stopping. The mine ventilation is performed by a suction method. Before the application of  $SF_6$  the following data were determined by the last measurement of the ventilation network and pressure imagery:  $Q_V = 20,55 \text{ m}^3/\text{s}$ ,  $\Delta p = 13,39 \text{ Pa}$ .

The first sampling of the air mass from the sampling site was made on 2.7.2007 at the barometric pressure of 1010.0 hPa (sample tube No. 84) and the second sampling was made on 5.7. 2007 at the barometric pressure of 1004.0 hPa (sample tube No. 92), see Table 1.

**Table 1** Application of  $SF_6$  - Frenštát Mine

Application of $SF_6$ Frenštát Mine - 29.06.2007				
Date and Time of Analysis	Sampling Site	Sampling Number	Concentration [ppb]	Sampling Date
5.7.2007 16:45-17:00	Borehole	84	0	2.7.2007
	Borehole	92	0	5.7.2007

The analysis of samples was performed using the gas chromatograph DANI GC 1000, infra-red analyzer and paramagnetic analyzer on 5.7. 2007. Presence of gas  $SF_6$  was not proved.

Next samplings of air mass were performed on 13. and 15.7. 2007 at the barometric pressures of 1,017.0 and 1,020.0 hPa (sample tubes No. 3, 84, 26 and 92). Assessment of the analyses from the day 16.07.2007 did not prove the presence of  $SF_6$  in the sample taken on 13.07.2007 (sample tubes No. 3, No. 84), however in the obtained samples from the day 15.07.2007 the presence of  $SF_6$  was determined. (Table 2).

**Table 2** Results of performed analysis of dry air sample

Analysis of Dry Air Sample - 16.07.2007									
Sampling Date	Sampling Time	Sample Tube Number	Sampling Site	Analysis Date Chromatograph	Analysis Time Chromatograph				
13.7.07	16:00	3	Frenštát	16.7.07	8:00				
13.7.07	16:00	84		16.7.07					
15.7.07	15:00	26	Frenštát	16.7.07	8:20				
15.7.07	15:00	92		16.7.07					
Results of Analysis - 16.07.2007									
$CO_2$ [‰]	$CH_4$ [‰]	$O_2$ [‰]	$CO$ [ppm]	$H_2$ [‰]	$SF_6$ [ppb]	Ethane [ppm]	Propane [ppm]	Iso- -butane	Propylene [ppm]
0,2	4,6	20,0	0	-	0	147,6	82,1	1,9	0
					0				
0,2	16	17,5	0	-	0,34	735,3	583	20,5	0
					0,34				

Considering the depth of discharge of  $SF_6$  (1,042 m below the surface – 590m below sea level Baa, south side of the shaft No. 5) and the distance between the borehole mouth and the shaft No. 5, by a simple calculation we get the direct distance covered by the applied gas from the discharge site to the sampling point at the mouth of the borehole NP 546 (+460 m above sea level Baa). This length is 1,390 m.

On the gas chromatograph the  $SF_6$  quantity of 0.345 was found out in the taken air-mass sample.

On 20. and 21.07.2007 air mass was subsequently sampled from a sampling point around the borehole No. NP 546 in the Trojanovice locality at the barometric pressure of 1,015.0 and 1,013.0 hPa. The results of the analysis of air mass samples performed on 23.07.2007 again demonstrated the presence of  $SF_6$  in the values of 0.3 ppb in the sample tubes No. 27, 49 and 0,4 ppb in the sample tubes No. 3 and 16 in both days, when the samples were taken. (Table 3).

**Table 3** Results of performed analysis of a dry air mass sample

Analysis of Dry Air Sample - 23.07.2007										
Sampling Date	Sampling Time	Sample Tube Number	Sampling Site	Analysis Date Chromatograph	Analysis Time Chromatograph					
20.7.07	15:00	27	Frenštát	23.7.07	8:00					
20.7.07	15:00	49		23.7.07						
21.7.07	15:00	3	Frenštát	23.7.07	8:20					
21.7.07	15:00	16		23.7.07						
Results of Analysis - 23.07.2007										
$CO_2$ [‰]	$CH_4$ [‰]	$O_2$ [‰]	$CO$ [ppm]	$H_2$ [‰]	Ethane [ppm]	$SF_{6,5}$ [ppb]	Propane [ppm]	Iso-butane	Propylene [ppm]	Butane [ppm]
0,1	19	16,8	0	-	575,7	0,3	240,4	21,3	0	12
						0,3				
0,2	8,1	19,1	0	-	156,3	0,4	59,2	5,2	0	1,9
						0,4				

On 29.7 2007 two air mass samples were taken from the above mentioned sampling point at the barometric pressure of 1014.0 hPa and the evaluation of the samples was carried out on 30.07.2007. In the sample tube No. 3 the presence of  $SF_6$  in the quantity of 0.1 ppb was proved. The values of gas analysis of the sample tube No. 165 the presence of  $SF_6$  did not prove (Table 4).

**Table 4** Results of analysis of a dry air sample

Analysis of Dry Air Sample - 30.07.2007									
Sampling Date	Sample Number	Tube	Sampling Site	Analysis Date Chromatograph			Analysis Time Chromatograph		
29.7.07	3		Frenštát	30.7.07			8:30		
29.7.07	165		Frenštát	30.7.07			9:15		
Results of Analysis - 30.07.2007									
$CO_2$ [‰]	$CH_4$ [‰]	$O_2$ [‰]	$CO$ [ppm]	$SF_6$ [ppb]	Ethane [ppm]	Propane [ppm]	Iso-butane	Propylene [ppm]	Butane [ppm]
0,1	17,5	17,3	1	0,1	2151,5	246	14,8	0	22,3
-	-	-	-	0	-	-	-	-	-

The last two samplings of air mass were performed on 12.08.2007 at the barometric pressure of 1,009.0 hPa (sample tube No. 27,44). The evaluation of the performed analysis did not prove the presence of gas  $SF_6$  (Table 5).

**Table 5** Results of analysis of dry air sample

Analysis of Dry Air Sample – 13.8.2007									
Sampling Date	Sample Number	Tube	Sampling Site	Analysis Date Chromatograph			Analysis Time Chromatograph		
12.8.07	27		Frenštát	13.8.07			9:30		
12.8.07	46		Frenštát	13.8.07			9:45		
Results of Analysis – 13.8.2007									
$CO_2$ [‰]	$CH_4$ [‰]	$O_2$ [‰]	$CO$ [ppm]	$SF_6$ [ppb]	Ethane [ppm]	Propane [ppm]	Iso-butane	Propylene [ppm]	Butane [ppm]
0,1	3,7	20,0	2	0	20,9	27,4	1,5	3,3	0
-	-	-	-	0	-	-	-	-	-

With regard to the way of the borehole No. NP 546 liquidation (cementing in full profile and length), the communication between the borehole mouth and the discharge point behind the stopping H6 can be explained either by the disruption of cement filling of the borehole or by the communication channels in the immediate vicinity of the well bore. The behaviour of values found out in time forecloses the migration of the gob atmosphere from productive carbon through the cover up to the surface around the borehole NP 546, which depends on the development of atmospheric pressure and parameters of the main fan at the Frenštát Mine ( $\Delta p = 13.39 \text{ Pa}$ ,  $Q_V = 20.55 \text{ m}^3 \cdot \text{s}^{-1}$ ). The mentioned migration demonstrably occurs, although the Frenštát Mine is ventilated by a suction method.

At the shutdown of the main fan of the Frenštát Mine an increase of migration outputs of the mine atmosphere with carbon gases can be assumed.

## 7 CONCLUSION

$SF_6$  tracer gas showed to be a useful and versatile tool for studying the ventilation network of mine. The United States Bureau of Mines successfully utilized  $SF_6$  for measurements and identifications of ventilation problems, as leakage of air mass through old stoppings, doors and cracks; mine air recirculation caused by its escape and by cooling equipment; air flow rate in airways of large diameters and low speeds; where the degree of air exchange in ventilated areas is insufficient. Simplicity and accuracy of the measurement technique by the

tracer gas when investigating the ventilation network, especially where conventional methods had failed, have resulted in a serious consideration of the adoption of tracer gas as a standard tool for examining ventilation issues.

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- [6] PROKOP, P., KOUDELKOVÁ, J.: 35/L3 Vědecko-výzkumná podpora významného posunu bezpečnosti při neřízeném výstupu stařínné atmosféry vycházející z řešení zbytkové plynodajnosti a plynonosnosti utlumovaných a opuštěných dolových partií, Dílčí zpráva, 2007, Ostrava.

#### RESUMÉ

Použití  $SF_6$  jako detekčního plynu může být efektivním prostředkem řešení výše uvedených problémů větrání. BÚ USA se zabývá vyhledáváním těchto problémů a jejich řešení včetně větrných programů. Za účelem použitelnosti detekčního plynu  $SF_6$  byly báňským úřadem uskutečněny na několika dolech různé studie.

Byl zkoumán proces uvolňování  $SF_6$  do důlní atmosféry. Hlavním problémem se ukázalo v neúplné míšení hustého  $SF_6$  s důlním ovzduším ve větrní chodbě s nízkým objemovým průtokem větrů. Při vyšších rychlostech proudění větrů tento problém se směřováním nenastal.

Jedno z důležitých použití indikačního plynu slouží k měření objemového průtoku větrů v chodbách, kdy pro standardní měření je průřez příliš velký anebo rychlost je příliš nízká. Experiment tohoto typu byl proveden v pensylvánském vápencovém dole s vertikálním přirozeným tahem vtažnou jámou asi 21,33 m hlubokou a 6,1 m v průměru. Jáma přivádí vtažné větry z povrchu dolů do chodby o průřezu cca  $74,32 \text{ m}^2$ . Pro použití měření rychlosti větrů anemometrem byl průřez příliš velký a rychlost příliš nízká. Proto bylo použito metody měření technikou indikačního plynu.