VERSATILE INFRARED LASER SOURCE FOR LOW-COST ANALYSIS OF GAS EMISSIONS

DELIVERABLE D3.3

Power, beam quality & spectral properties of DFG

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University of Southampton	ORC	United Kingdom
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1. INTRODUCTION

The main objective of the workpackage WP3 for months 1 – 12 was the implementation of a mid-IR source, based on difference frequency generation (DFG) in an orientation-patterned GaAs (OP-GaAs) crystal.

Efforts of the consortium participants during the first six months were focused on

- Precise understanding of the requirements from each participant in the design of all the subparts
- Chose of relevant molecular gas species and absorption lines (D3.1) for demonstration of the first spectroscopic sensor (D4.2).

Besides, the preliminary experiment on DFG in OP-GaAs crystal has been carried out during months 1 - 6. Narrowband tunable lasers at 1.56 µm and 3.6 µm were mixed to get sub-µW DFG output at 2.76 µm. Main objectives of this experiment were :

- Testing and characterization of the first OP-GaAs crystal test-samples.
- Testing of the DFG source design options
- Comparison of the theoretical predictions for the DFG source (output power, bandwidth, wavelengths corresponding to the optimal QPM conditions) with obtained experimental results.

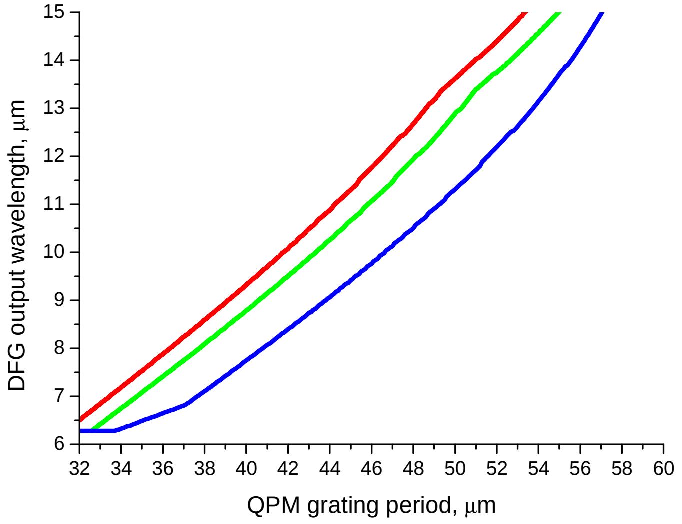

Figure 1 : DFG tuning as a function of OP-GaAs period.

The project work-plan assumes implementation of the mid-IR DFG source based on a commercial erbium doped fiber amplifier EDFA seeded by a narrowband diode laser and a first version of the thulium (Tm) doped DFB fiber laser from ORC.

Standard tuning range for commercial EDFA is 1540 – 1570 nm, while the gain curve in Tm-doped fibers makes it possible to get emission wavelengths from 1740 to 2040 nm. The Figure 1 :shows potential spectral coverage of the DFG source based on mixing of Er- and Tm-doped fiber lasers. The output wavelength of the DFG source is shown as a function of the QPM grating period in OP-GaAs crystal. Blue curve is the minimum DFG output wavelength (for the crystal temperature of 20℃). Green curve is the maximum DFG output wavelength (for maximum crystal temperature of 100°C). Red curve is the maximum DFG output wavelength (for maximum crystal temperature of 175℃).

For reasons related to material growth, it was initially planned to restrict the first samples to periods around 60 µm. Due to recent progress, this figure can reasonably be lowered down to around 40 µm. As a result, the mid-IR wavelengths generated by DFG can be extended from around 15 µm down to around 7 µm, thus making easier optical detection and widening the list in which the first test gas can be selected.

Potential gases of interest have been investigated in the wavelength range, which can be covered using the DFG source. The main selection criterion for a particular gases and absorption lines was a trade-off between relevance for combustion and emission control, line strength, and interference with other gaseous species. Selected species, line positions and detection limits are listed in table 1.

Species	Line position µm	Line strength cm^{-1}/molec.	Detection limit 10^{-5}*1m
NH_3	~8.9	4E-20...9E-20	2.5 ppb
SO_2	~8.9	1E-21	49 ppb
H_2O_2	~7.7	1E-20	10 ppb
CH_4	~7.7	3E-20	10 ppb
NO_2	~6.2	3E-20...1E-19	1.4 ppb

Table 1: Potential gases of interest for DFG based mid-IR source capable of selective gas detection

(T = 296 K, p = 1 atm).

Key choices for the DFG source design were based on a compromise between the specifications of Tm-fiber laser and OP-GaAs crystal, suited to realize a simple and powerful enough setup in the wavelength range corresponding to the specified lines position. As long as the expected DFG source can yield wavelengths above 7 µm, our attention was focused on ammonia and methane because they can be quite easily handled. This last option has finally been chosen. Thus the DFG source is based on a following nonlinear mixing scheme:

$$1.55\ \mu m + 1.94\ \mu m \rightarrow 7.67\ \mu m, \text{ with OP-GaAs period of } 38.6\ \mu m$$

2. PRELIMINARY EXPERIMENT ON DFG IN OP-GAAS CRYSTAL (MONTHS 1-6)

For a “quick-start” of the DFG source development a preliminary experiment on DFG in orientation patterned GaAs crystal (OP-GaAs) has been carried out during months 1 - 6. The test-sample of the OP-GaAs crystal with the grating period of 27.6 µm was provided by TRT. The 0 illustrates DFG spectral range achievable for this grating period at the room temperature if the EDFA is used as a primary DFG pump. This estimation was carried out using GaAs dispersion data from [1]. As follows from the figure, the EDFA output can be mixed with 2.5 – 4 µm radiation, which corresponds to the tuning range of Nd:YAG pumped PPLN OPOs.

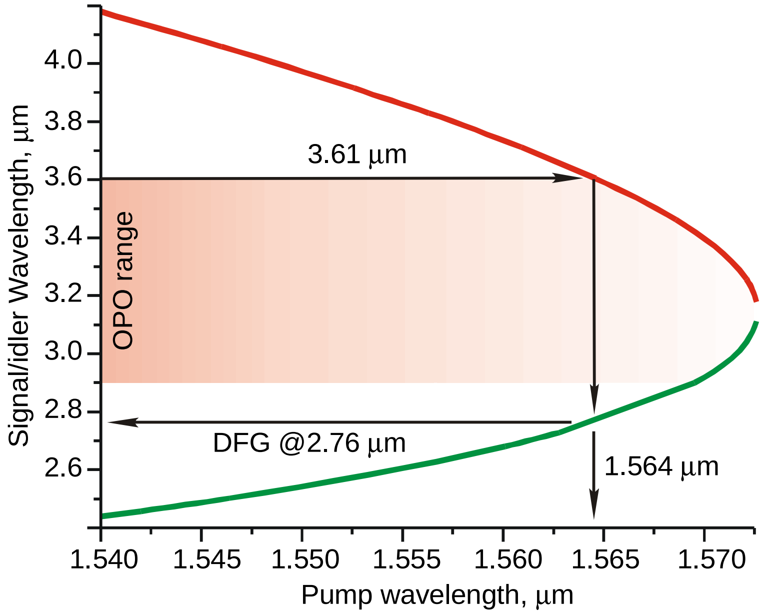

Figure 2 : DFG tuning as a function pump wavelength.

The tuning range of the single-frequency CW PPLN OPO, available in HHUD, is shown in the 0by the red filling. The OPO is equipped with a 19-gratings PPLN crystal and uses a dual-cavity PR-SRO design [2]. For the DFG experiment the OPO was tuned to the maximum wavelength of 3.61 µm. With a corresponding EDFA wavelength of 1.564 µm the resulting DFG wavelength of 2.76 µm was estimated (black arrows in the 0). At the 1.064 µm pump power of 700 mW the OPO output power at 3.61 µm was about 60 mW.

A schematic of the experimental setup is shown in the Figure 3 :. The pump source was a 1-W EDFA provided by TRT. A single-frequency tunable external cavity diode laser (ECDL) (Agilent 8169A) with 1.51 – 1.64 µm tuning range and 3 mW maximum output power was used as EDFAs seed. The idler source was the PPLN OPO, described above. Polarizations of both sources were set to be parallel to the [100] growth direction of the OP-GaAs crystal. The 9.5 mm long crystal was uncoated and the crystal’s patterned region thickness was about 0.5 mm. The OP-GaAs crystal was mounted on a Cu heat sink.

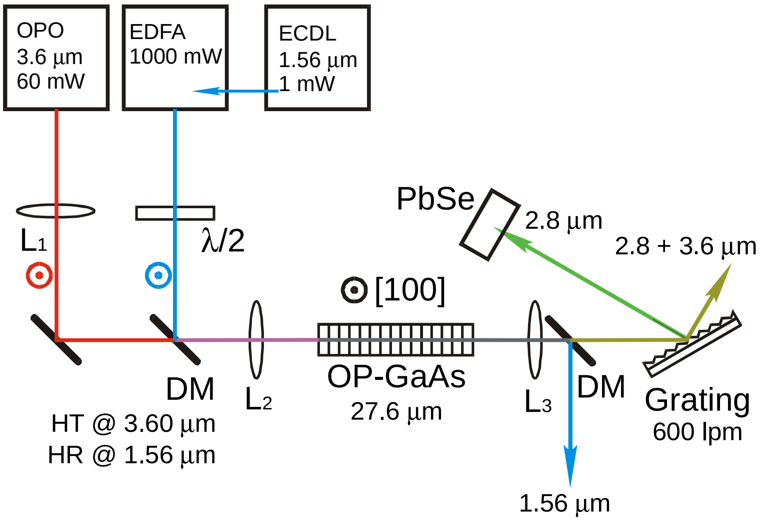

Figure 3 : Preliminary DFG setup.

Experiments were carried out at the room temperature (22 ^{O}C). No active temperature control of the crystal was used. Pump and idler beams were combined using a dichroic mirror and focused at the OP-GaAs crystal by f = 150 mm uncoated CaF2 lens. Waist diameters of 80 µm and 230 µm were measured for the EDFA and OPO beams respectively. A second lens (L3) collimated the output beams and a second dichroic was used to cut off the high power pump radiation. Radiation at the DFG signal wavelength (2.76 µm) was separated from residual pump and idler by a 600 lines per mm diffraction grating and detected by an uncooled PbSe detector (ThorLabs PDA20H) using a lock-in amplifier.

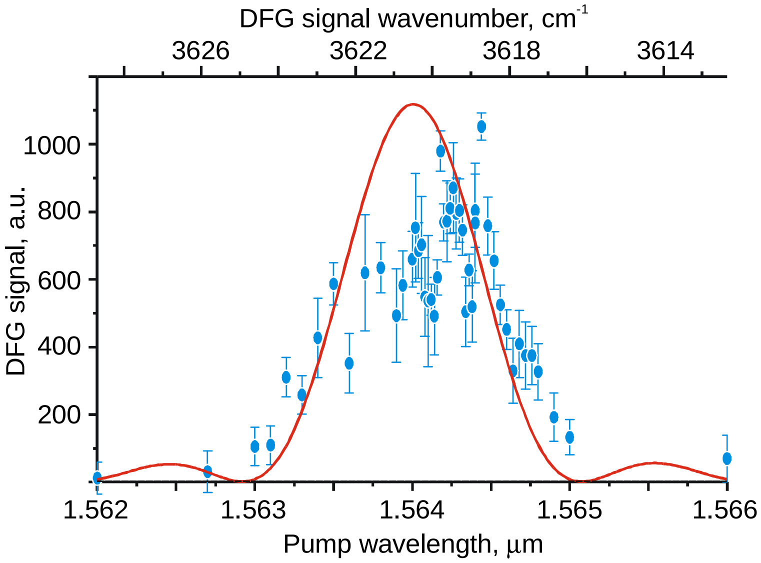

Figure 4 : Tuning results.

The DFG signal tuning curve was recorded by pump wavelength tuning while the idler (OPO) wavelength was fixed at 3.608 µm. The detected DFG signal versus pump wavelength is shown in the Figure 4 :by blue circles and the theoretical curve, which was calculated using literature data on GaAs crystal dispersion, is shown by the red curve. As can be seen, positions of measured and calculated peaks are almost coinciding. The measured bandwidth (FWHM) is slightly higher than the theoretical estimate (4 cm^{-1}). Such an agreement demonstrates a high uniformity of the OP-GaAs sample. The deviation of the tuning curve from the sinc^2 behavior may be partly explained by instabilities of the OPO output wavelength and power during the measurement cycle[1] and by etalon effects on uncoated crystal surfaces.

The maximum output power of the DFG setup can be estimated using the following formula [3]:

$$P_s = \eta_0 L P_p P_i$$

$$\eta_0 = \frac{4}{\pi} \frac{\omega_i^3 \omega_p \omega_s d_{eff}^2}{\left(n_p \omega_p + n_s \omega_s\right)^2 c^4 \varepsilon_0},$$

In this formula, L is the crystal length; $P_{p,i,s}$, $n_{p,i,s}$, $\omega_{p,i,s}$ are respective powers, refractive indices and frequencies of the pump, idler and signal beams; $d_{eff} \approx$ 100 pm/V is the effective nonlinear coefficient, c is the speed of light and ε_0 is the free space permittivity. The expression for the normalized efficiency η_0 [W^{-1}·m^{-1}] holds for optimal beam focusing, where the idler beam Rayleigh range $z_{0(i)} = \pi w_i^2 / \lambda_i$ is equal to the half of the crystal length ($w_i^{(opt)}$ = 75 µm).

Taking into account losses in the beam combining system and 30% reflection losses on uncoated crystal ends, the upper estimate for the DFG output is about 30 µW. The detected DFG signal was in the sub-µW range. This difference can be mostly explained by non-ideal pump beam focusing and beam overlap in the crystal. Besides, non-ideal grating periodicity of OP-GaAs test-sample (missing domains) and considerable parasitic losses of the test crystal sample (>0.1 cm^{-1}) has reduced the nonlinear conversion efficiency.

The following conclusions were made regarding the further development of the mid-IR DFG source based on Er- and Tm- doped fiber lasers:

1. Very good agreement between the theoretical predictions and the experimental results (in terms of bandwidth and exact wavelengths corresponding to the optimal QPM conditions) confirm correctness of models and of the software, used for simulations
2. The beam combining and focusing system has to be improved (use of higher quality dichroic mirror for the beam combining; the pump and signal beams have to be focused and adjusted independently
3. Use of a combination of the dichroic mirror and the diffraction grating for beam separation is not efficient due to the low extinction of the standard dielectric coated optics, limited diffraction efficiency of the grating, and a considerable level of the background scattering from the grating
4. Assuming the few hundreds of mW power lever for the first version of the Tm-doped DFB fiber laser, the use of the high-power EDFA may be essential to achieve the mid-IR output power, high enough for the selective gas detection
5. Optical isolation of the pump and signal lasers from the DFG setup may be required, although, compared with bulk narrowband sources, the DFB fiber lasers are less sensitive to the parasitic back-reflections and scattering.

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[1] The OPO source was not optically isolated from the DFG setup and the instabilities of the OPO output were due to parasitic reflections from uncoated lens optics and the crystal.

3. COMPACT SETUP FOR DFG BETWEEN TWO FIBER COUPLED LASER SOURCES CAPABLE OF SELECTIVE GAS DETECTION

3.1. Inputs from ORC and TRT and high-power EDFA

The source of the “signal” radiation for the DFG setup was a Tm-doped DFB fiber laser, provided by ORC. Main parameters of the Tm-doped fiber laser source a summarized in the table 2

Pump source	Core pumping by the broad band Er-doped fiber laser
Maximum output power in “forward” direction	0.25 W *)
Maximum output power in “backward” direction	0.05 W *)
Central wavelength	1942.9 nm
Fine tuning :	≤ 0.5 nm (by PZT with 20 μm travel)
Polarization	Two orthogonally-polarized modes with 600 MHz shift
Input/output fiber termination	Angle-cleaved bare fiber

Table 2: Parameters of the Tm-doped DFB fiber source

The Tm-doped fiber laser output power measured in HHUD was approximately 20% lower than the power level achieved in ORC. This can be explained by some degradation of the bare fiber ends. Besides, different types of aspheric optics were used in HHUD for the pump beam focusing and the output beam collimation.

The source of the “pump” radiation for the DFG setup was a commercial high power Er-doped fiber amplifier (Keopsys KPS-STD-BT-C-40-SLM). The EDFA was seeded by the single-frequency tunable external cavity diode laser (ECDL) (Agilent 8169A) with the maximum output power 3 mW Main parameters of the ECDL seeded EDFA source a summarized in the table 3

Output power	≈ 10 W
Output power stability	≈ 1 %
Tuning range:	1540-1570 nm with sub-pm precision
Frequency stability	< 100 MHz
Polarization	90% linear
Output fiber termination	1-m long pigtail with a standard collimator; output beam ∅2 mm, M2 ≈ 1.1
Output isolation	Optional

Table 2: Parameters of the EDFA

The OP-GaAs crystal for DFG setup was provided by TRT. The crystal has two patterned regions with QPM grating periods of 38.6 and 53.8 μm. Thus, the crystal can be used as for DFG experiments as well as for the future Tm-doped fiber laser pumped OPO development. Compared with the first test-sample of the crystal (see section 2), the new OP-GaAs sample is 3 times longer (33 mm vs. 9.5 mm) and has much lower level of the parasitic losses (0.01–0.05 cm^{-1} vs. 0.1 – 0.15^{-1}). The sample is uncoated, which introduces a 30% parasitic reflectance at each side of the crystal.

The thickness of the OP crystal layer was slightly non-uniform: ≈ 400 μm on the one crystal end and ≈ 550 μm on the other end. Naturally, the "thin" end of the crystal was used as an input port for the short-wavelength pump/signal beams and the "thick" end was used as an output port for mid-IR (8-μm) idler beam.

Due to high index of refraction of the material ($n_{GaAs} \approx 3.3$), the focusing of the pump/signal radiation (1.55/2 μm wavelength) can be rather tight. The focusing parameter ξ can be as high as 2.8 that correspond to the beam waist radius of 30 μm at the 2 μm wavelength. The optimal $\xi = 2.8$ beam focusing can be achieved for the 8-μm idler wavelength as well. However, to prevent possible aperture effects, we have decided to use confocal ($\xi = 1$) beams focusing scheme, which cause just insignificant (10%) reduction of the nonlinear conversion efficiency.

3.2. DFG source arrangement

Schematic of the mid-IR DFG source is shown in the Figure 5 :. The source consist of:

- Broadly tunable EDFA source (described above)
- Tm doped laser setup (described above)
- Optional units for optical isolation and polarization control
- Pump/signal beam combining system (dichroic mirror DM)
- Pump/signal beam focusing system (L1/L2 lens optics)
- OP-GaAs crystal unit, which includes multi-axis crystal mount, crystal oven, and electronics for crystal temperature control and tuning
- Output beam collimation system (ZnSe mid-IR lens optics)
- High-extinction-ratio beam separation system (≥10000:1 extinction at 1.55 μm wavelength is required to achieve acceptable signal-to-noise figure at the detector)
- Mid-IR detection system, which includes pyroelectric or semiconductor detector, and necessary electronics
- Wavelength tuning and control system (see section 3.3), which includes PZT driver for the Tm-doped DFB laser scanning. Optionally, the DFG setup can be equipped with wavelength measurement system for precise adjustment and tuning of the Tm-doped DFB laser wavelength. The wavelength measurement system can be based on a laser wavelength meter or a reference gas cell. Another option is the use of a reference cavity for active stabilization of the Tm- laser output wavelength.

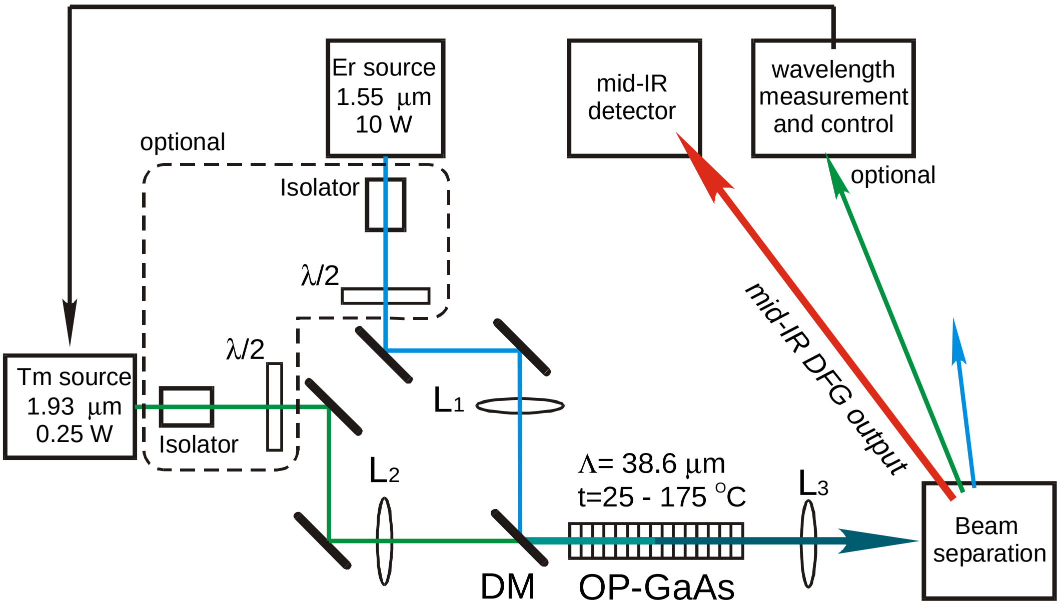


Figure 5 : Fiber DFG setup.

The DFG source layout-drawing is shown in the Figure 6 :.All the optical components of the DFG source are arranged on a standard optical breadboard. Approximate dimensions of the optical part of the setop are 450W)×600(L)×200(H) mm. Most part of the optomechanis are standard components. Some adapters, the crystal oven and oven controller were fabricated in HHUD's mechanical and electronics workshops. Most part of the optics is custom coated. The first version of the Tm-doped DFB laser source has rather short bare-ended output fiber. Therefore, additional free-space optics was used for collimation and delivery of the Tm-laser beam to the DFG setup, while the Figure 6 :shows the final setup with the fiber ports for both Tm- and Er-doped fiber lasers.

Our preliminary experiments have shown that the use of standard dielectric coated optics or grating is not an efficient solution for the high-extintion-ratio beam separation system (see section 2), while the use of enhanced dielectric coated optics does not seems cost-efficient. Therefore, we have desided to use a custom made prism for separation of the weak idler beam at 8 µm from high power pump/signal beams at 1.5/2 µm.

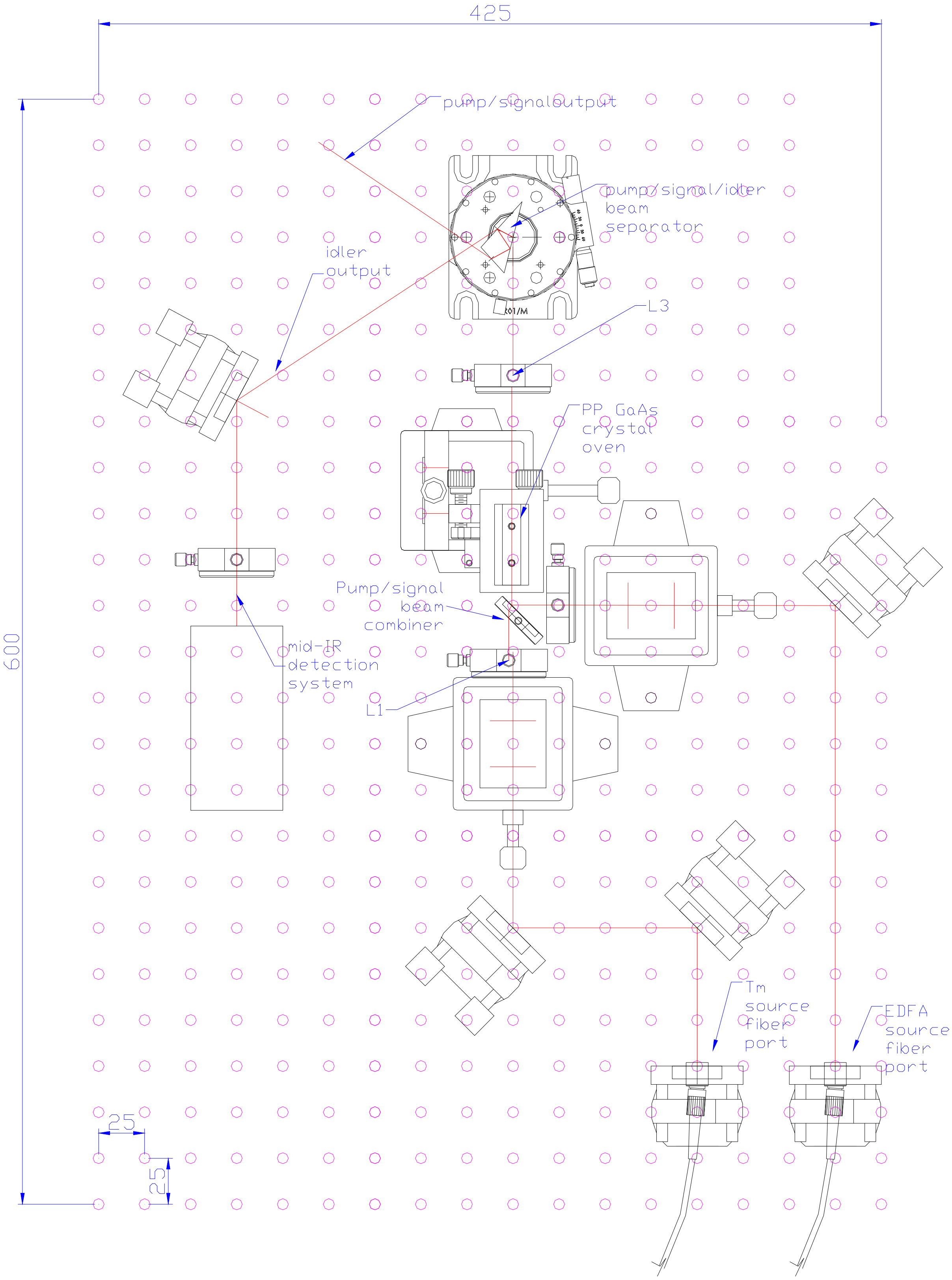


Figure 6 : Layout of the fiber DFG setup.

3.3. Frequency control and tuning

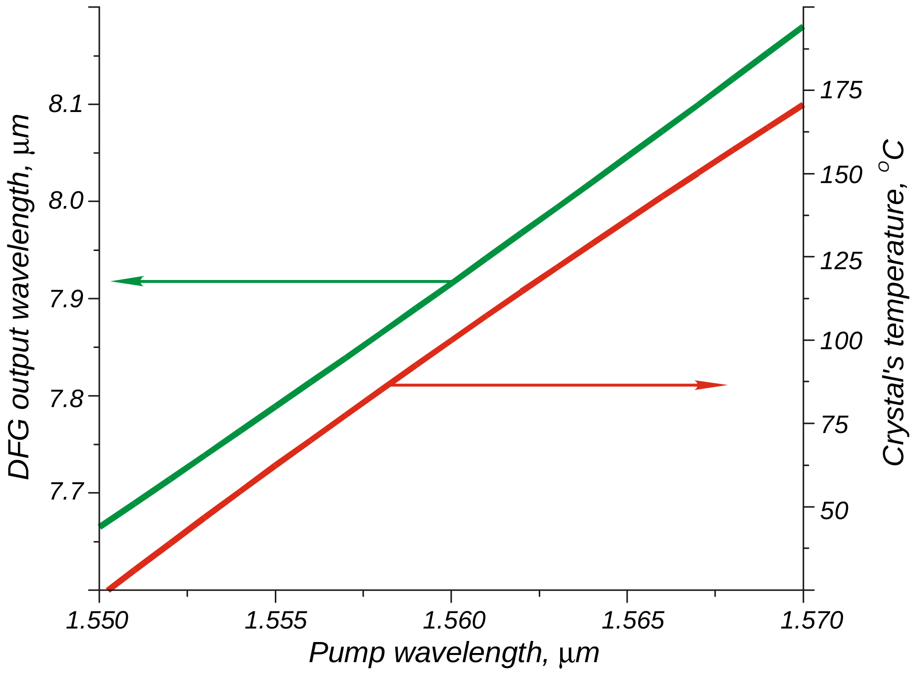


Figure 7 : Fiber DFG tuning.

Two laser sources are used for DFG setup pumping: broadly tunable EDFA with 1540 – 1570 nm range (DFG pump) and Tm- doped DFB fiber laser with central wavelength of 1942.9 nm and tuning range of 0.5 nm (DFG signal). The spectral coverage of the DFG source, for these pumping conditions and OP-GaAs crystal with QPM grating period of 38.6 µm, is shown in the Figure 7 :. The green line shows the DFG source output wavelength as a function of the EDFA pump wavelength and the red line shows the crystal temperature corresponding to the optimal QPM conditions. As can be seen, the tuning range of the DFG source is rather broad (7.6 – 8.2 µm), which allows detection of a variety of gas species using the same device.

Two frequency tuning options are implemented in the DFG setup:

- "Slow" broadband tuning, by simultaneous tuning of the EDFA wavelength and the crystal temperature, to adjust the setup on a particular gas absorption line
- "Fast" scanning within a chosen absorption line bandwidth, by the Tm-doped DFB fiber laser tuning, and at constant temperature of the crystal.

The "slow" tuning may be automated by a computer control of the EDFA's seed laser (via GPIB interface) and of the oven temperature controller. However, a manual adjustment of the pump wavelength and of the temperature is used at the moment.

The "fast" scanning of the DFG output wavelength is also an "automation-ready". At the moment, this tuning channel is controlled by programmable function generator, which allows to adjust the tuning range (within 0.5 nm) and the repetition rate of the scan-cycles.

The output wavelength of the EDFA (DFG pump) can be defined and hold with a sub-pm precision; therefore, the DFG output wavelength can be measured by measuring of the Tm-doped DFB fiber laser wavelength. This can be done using a laser wavelength meter, or by using a reference gas cell.

3.4. Output power estimates and scale-up options

The output power of the mid-IR DFG source can be estimated using formulae, given in the section 2. The upper estimate for the 3-cm long AR/AR coated OP-GaAs crystal and 10/0.25-W pump/signal laser power is about 2-mW. The upper estimation for the uncoated PP-GaAs crystal is about 0.7 mW (the 3 times reduction is due to the 30% Fresnel reflectivity at all wavelengths). In our opinion, more realistic estimation for DFG output power is 0.3 – 0.5 mW (depending on a crystal quality and beam matching conditions.

As it was mentioned above, the AR coating of the crystal allows 3-times enhancement of the mid-IR output power. Another straightforward way to scale-up the DFG source output is an increase of the pump and signal lasers power. This option can be implemented using a high-power Tm-doped fiber MOPA (deliverable D1.2).

Use of the resonantly enhanced DFG technique. This consists in resonating the single-frequency Tm- laser radiation in a ring cavity, while the Er-laser radiation is not resonated and pass through the nonlinear crystal once only. This option, although more complicated in realization, is closer to the final OPO setup. The enhancement factor $G \approx 1/\alpha \approx 10$ may be expected for a cavity round trip losses $\alpha = 10\%$.

Another interesting option, which is relatively simple in realization, is DFG in a cascade of OP-GaAs crystals. This approach has been recently tested in HHUD using the cascade of two PPLN crystals; 3.5 times enhancement of the nonlinear conversion efficiency has been demonstrated.

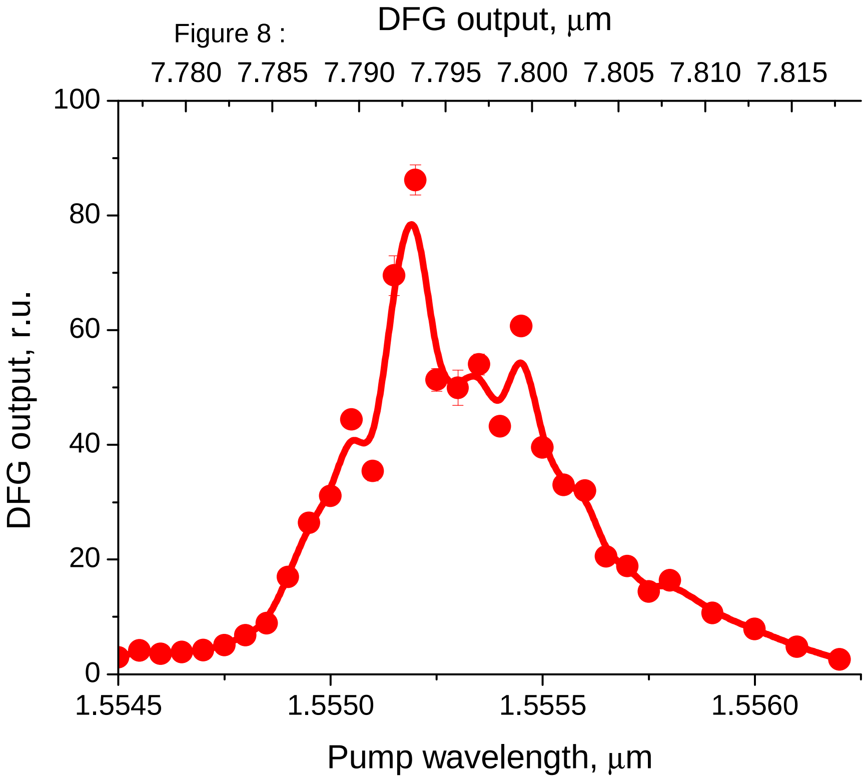
4. POWER, BEAM QUALITY AND SPECTRAL PROPERTIES OF THE DFG

The first experimental demonstration of the mid-IR source based on the DFG between Er- and Tm-doped fiber lasers has been carried out using a simplified beam separation system, which is shown in the Figure 8. The high power Er- laser beam and medium power Tm- laser beam were combined using the dichroic mirror DM and focused in to the OP-GaAs crystal. The output beams were collimated by the lens L3. The dichroic mirror DM1 with HR at the Er- laser wavelength and 80% transmission at the mid-IR output wavelength was used to separate the high power 1.55 μm radiation. The medium power Tm- laser radiation and residual Er- laser radiation were blocked by longwave-pass optical interference filter (SPECTROGON LP-7000, provided by TRT). The DFG output signal at 8 μm wavelength has been detected by a pyroelectric detector.

This “straightforward” beam separation system is far from ideal in terms of losses (the 50% transmission at 8 μm was estimated). However it is much more simple in arrangement, and allows faster and easier optimization of the beams overlap and focusing in the non-linear crystal.

A DFG output signal has been easily detected using described optical set up. The first example of the DFG tuning curve is shown in the Figure 8 :. The tuning curve was recorded by “pump” (Er-laser) wavelength tuning while the “signal” (Tm- laser) wavelength was fixed. The Er-laser wavelength, which corresponds to the maximum DFG signal was shifted from the expected value for about 0.5 nm. This may be explained by a heating of the pumped channel in the OP-GaAs crystal by high power Er-laser radiation. Measured value of the shift corresponds to the 4 - 6°C heating of the pumped channel with respect to the crystal’s oven temperature.

Figure 8 :



The non-uniform shape of the tuning curve may be partly explained by the etalon effects on uncoated surfaces of the crystal and by interference effects on elements of the beam separation system. We expect to achieve more uniform tuning behavior in fully optimized DFG set-up.

The measured signal on the pyroelectric detector corresponds to approximately 100-µW power level of the mid-IR DFG output. We expect considerable improvement of the DFG output power after accurate optimization of the crystal orientation, beams overlap, and beams focusing.

Another important result of the first experiment with DFG source is the successful test of the OP-GaAs crystal under high-power near-IR laser irradiation. No visible degradation of the crystal has been observed during long-term 10-W CW laser pumping (approx. 5 hours). Our estimate for the heating of the pumped channel in the OP-GaAs crystal confirms the low level of the parasitic absorption in the material.

5. CONCLUSIONS

Active collaboration between all partners has enabled to reach the important milestone in the project work-plan. The mid-IR source capable of selective gas detection in 7.6 – 8.2 µm spectral range has been developed. This source is based on the difference frequency generation (DFG) between a high-power Er-doped fiber amplifier (commercial) and a medium-power Tm-doped DFB fiber laser (first version of the Tm- laser from ORC, D1.1).

Implementation of the DFG source has been carried out on an “automation-ready” basis with regard to the development of the First gas sensor demonstrator (D 4.2)

Preliminary characterizations of this setup have shown promising results. More experiments are under way to quantify the nonlinear gain in OP-GaAs crystal, of importance for the development of the final OPO-based mid-IR source. As an alternative option, it has also been shown that the output power of the DFG source can potentially be further scaled-up by a factor of 3 - 30, depending on choice of the power enhancement option.