Towards the realization of atom trap trace analysis for $^{39}$Ar

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Abstract. We present our study of the realization of atom trap trace analysis for $^{39}$Ar, an ultra-sensitive detection method for rare isotopes based on laser cooling. We report on the experimental determination of the hyperfine spectrum of the relevant cooling transition. A high-intensity, optically collimated beam of metastable argon atoms has been set up, and fluorescence detection of single $^{40}$Ar atoms in a magneto-optical trap is realized. The deduced efficiencies of each stage lead to an expected $^{39}$Ar count rate of six atoms per hour in the final setup.
1. Introduction

The radioactive isotope $^{39}$Ar ($T_{1/2} = 269$ a, [1]) has enormous potential in environmental physics for bridging the dating gap between 50 and 1000 years [2]. This is the timescale of many interesting aspects connected with water cycles, such as ocean circulation, continental glaciers, water resource management and, generally, the anthropogenic influence on the environment over the last millennium. The dating gap arises from the lack of any reliable dating tracer available for this time window. Although ultra-sensitive methods for $^{39}$Ar detection are already available [3]–[5], they require very involved experimental efforts. The application of a standard dating tracer may become feasible by realizing atom trap trace analysis (ATTA) for this isotope, a method that has been successfully demonstrated for rare isotopes of Kr [6] and Ca [7]. This method achieves a significantly higher selectivity than established methods by making use of the shift of optical transitions between isotopes and the comparatively small linewidths.

Several problems have to be dealt with in the course of setting up $^{39}$Ar-ATTA, one of them being the low isotopic abundance of $^{39}$Ar/Ar $\sim 10^{-15}$ in typical samples. The efficiency of the vacuum apparatus relies on the purity of the injected sample; hence, improved techniques for water degassing and gas separation need to be applied. The standard water sample in oceanography is 10 litres, which corresponds to $10^{20}$ argon atoms. This imposes demanding constraints on the apparatus, implying high throughput efficiency and sample recycling.

The high-throughput atomic beam apparatus features an intense metastable atom source. For these excited states, laser cooling is possible by using readily available diode lasers. This is used to reduce the transverse and longitudinal velocity of the atomic beam. In combination with a magnetic field, it allows for the trapping of atoms in a magneto-optical trap (MOT). Detection of the fluorescence light from the MOT helps in counting individual atoms.

We report on the crucial steps towards realizing such an atomic beam apparatus. One of these is the first spectroscopic measurement of the hyperfine structure (hfs) of the designated cooling transition in $^{39}$Ar, $1s_3 – 2p_9$. The experimental procedure and results are briefly presented in the corresponding sections.

Further focus is on the setting up of an intense, collimated beam of metastable argon atoms. Simulations were carried out to optimize the design parameters, and these theoretical predictions are in good agreement with the experimental findings for $^{40}$Ar. Another crucial step in the
Figure 1. The setup for Doppler-free saturation spectroscopy in $^{39}$Ar. We operate two counterpropagating laser beams of which one is frequency shifted by a double-pass AOM. The strong pump beam ($I = 3 \times I_{\text{sat}}$) enters the cell from the left side. The weak probe beam ($I = 0.7 \times I_{\text{sat}}$) is detected on a fast photodiode after passing through the spectroscopy cell. The Fabry–Pérot interferometer (FPI) is used as a precise frequency scale.

The framework of ATTA is the detection of single argon atoms in a MOT. The optimal parameters for atom detectability are derived experimentally and the resulting single atom signatures are discussed. In conclusion, we derive the expected count rate for $^{39}$Ar.

2. Spectroscopy

We access the hfs spectrum of the $1s_5-2p_9$ transition by means of modulation transfer laser spectroscopy with an external cavity diode laser at 811.754 nm in an isotopically enriched argon sample. The experimental setup is sketched in figure 1. The measurement setup and procedure are explained in detail in [8, 9].

The gas sample was extracted from potassium-rich mineral that has been irradiated in a neutron reactor leading to the production of $^{39}$Ar by $^{39}$K(n, p)$^{39}$Ar. The mineral also contains $^{40}$Ar from in situ $^{40}$K decay, leading to $^{39}$Ar/$^{40}$Ar $\sim 0.8$ as determined via mass spectrometry of a simultaneously irradiated sample.
2.1. Spectroscopic setup

A quartz glass cell containing the enriched sample is used for spectroscopy and a high-power radiofrequency (RF) discharge maintains the plasma necessary for population of the metastable state. The cell is thoroughly cleaned by following a special scheme to overcome the problem of outgassing during the discharge operation [8]. After the transfer of the isotopically enriched sample, the cell contains 3 nlSTP $^{40}$Ar and 2.5 nlSTP $^{39}$Ar at a total pressure of $\sim 2 \times 10^{-4}$ mbar.\(^4\)

The pump beam of the modulation transfer Doppler-free saturation spectroscopy [10] is frequency modulated by a double-pass acousto-optic modulator (AOM; modulation frequency 22 kHz and amplitude 10 MHz). The transmitted probe beam is detected with a photodiode and the signal is demodulated using a lock-in amplifier. The signal-to-noise ratio of the data is improved by additional averaging over many laser frequency scans. Nonlinearities in the laser frequency scan are compensated for by using the transmission peaks of an FPI as a precise frequency scale. The pronounced $^{40}$Ar peak is used as a reference for averaging the single scans and thus allows for compensation of laser drifts. This results in a spectroscopy signal, as shown in figure 2. Since the laser linewidth is $\sim 0.5$ MHz and the gas pressure is low ($<10^{-3}$ mbar), the predominant line broadening mechanisms are power and modulation broadening [9]. These effects result in a measured linewidth of 30 MHz, whereas the natural linewidth of the transition is 5.85 MHz [11].

\(^4\) nlSTP stands for nanolitre at standard temperature and pressure.

**Figure 2.** The hfs spectrum of $^{39}$Ar. Signal quality is increased by averaging over 3200 single scans. The resonances calculated from the fitted parameters are indicated by vertical lines. For clarity, calculated crossover resonances are shown not in the main figure but in the inset, where they are indicated by dotted lines.
2.2. Spectroscopic data

The data are fitted with hyperfine splittings according to the equation [12]

$$\Delta E = \frac{A}{2} C + \frac{B}{4} \frac{3C(C+1) - 2I(I+1)J(J+1)}{I(2I-1)J(2J-1)},$$

where $J$ is the total angular momentum and $I$ the nuclear spin. The magnetic dipole and electric quadrupole moments are expressed by the hyperfine constants $A$ and $B$, respectively. The Casimir constant is defined by $C = F(F+1) - I(I+1) - J(J+1)$, where $F = J + I$. With $I({}^{39}\text{Ar}) = +7/2$, $J(1s_5) = 2$ and $J(2p_9) = 3$ the metastable state splits up into five levels from $F = 3/2$ to $11/2$ and the excited state splits up into seven levels from $F = 1/2$ to $13/2$, as illustrated in figure 3. This leads to a set of 15 transitions according to selection rules and also 30 crossover resonances.
Table 1. Data for the constants of the $1s_5$ and $2p_9$ levels of $^{39}$Ar as estimated and determined from our data. The shift of $^{39}$Ar relative to $^{40}$Ar is calculated with an empirical formula, as explained in detail in [8]. The larger errors on the $B_s$ result from equation (1) being much less sensitive to $B$ than to $A$. The measured values agree with the calculated values within errors ($1\sigma$).

|       | $\Delta \nu$ (MHz) | $A_s$ (MHz) | $B_s$ (MHz) | $A_p$ (MHz) | $B_p$ (MHz) |
|-------|---------------------|-------------|-------------|-------------|-------------|
| Calc. | $-97.2 \pm 4.5^a$   | $-286.1 \pm 1.4^b$ | $118 \pm 20^b$ | $-133 \pm 14^c$ | $115 \pm 3^c$ |
| Meas. | $-95.0 \pm 0.4$     | $-287.15 \pm 0.14$ | $119.3 \pm 1.5$ | $-135.16 \pm 0.12$ | $113.6 \pm 1.9$ |

$^a$ Analysis using data from [13] and [14] for different lines in $^{36}$Ar, $^{38}$Ar and $^{39}$Ar.

$^b$ Calculated by Klein et al [13]: analysis using data from $^{37}$Ar.

$^c$ Estimated from the hfs constants of other noble gas isotopes.

Figure 4. Schematic illustration of the apparatus design for ATTA of $^{39}$Ar. Metastable argon atoms are produced in an RF-driven source and transversally cooled in a collimation stage. Subsequently, the atoms are slowed down and captured in the MOT, where their fluorescence light is imaged onto an optical fibre that is connected to an avalanche photodiode (APD).

The hfs constants $A_s$, $B_s$, $A_p$, and $B_p$ and the—mainly mass-induced—isotopic shift $\Delta \nu$ are deduced by fitting the corresponding line positions. These results are compared with calculated values in table 1. The lines derived from the fitted parameters are plotted in figure 2 and show very good agreement within experimental errors.

3. Apparatus

The apparatus design consists of the different stages depicted in figure 4. Maximizing the flux of metastable argon atoms into the MOT is crucial for efficient detection of $^{39}$Ar. Zeeman slowers and MOTs have been optimized by different groups and can be regarded as standard tools. The specifics to $^{39}$Ar-ATTA are the realization of an optimized source and a collimation stage, which will be discussed next.
### Table 2. Comparison of the different sources for the production of metastable atomic beams.

| Element | Source          | Excitation | Total throughput (atoms s⁻¹) | Metastable intensity (atoms s⁻¹ sr⁻¹) | Reference       |
|---------|----------------|------------|------------------------------|--------------------------------------|-----------------|
| He      | Supersonic     | dc         | $1.2 \times 10^{19}$        | $26.6 \pm 5.3 \times 10^{14}$        | [15, 16]        |
| He      | Supersonic     | Electron beam | $2 \times 10^{21}$         | $3 \times 10^{14}$                   | [17]            |
| He      | Supersonic     | dc         | $-$                         | $10^{14}$                            | [18]            |
| Ne      | Effusive       | dc         | $-$                         | $1 \times 10^{15}$                   | [19]            |
| Ne      | Supersonic     | dc         | $8 \times 10^{18}$         | $11 \pm 2.8 \times 10^{14}$         | [15, 16]        |
| Kr      | Effusive       | Microwave  | $1 \times 10^{17}$         | $7 \times 10^{14}$                   | [20]            |
| Kr      | Effusive       | Optical    | $3 \times 10^{17}$         | $3 \times 10^{14}$                   | [21]            |
| Kr      | Effusive       | RF         | $7 \times 10^{16}$         | $4 \times 10^{14}$                   | [22]            |
| Ar      | Supersonic     | dc         | $1.7 \times 10^{18}$       | $4.3 \pm 2.2 \times 10^{14}$         | [15, 16]        |
| Ar      | Effusive       | RF         | $2.4 \times 10^{18}$       | $6.0 \pm 1.5 \times 10^{14}$         | [15, 16]        |

#### 3.1. Source

A metastable argon beam can be produced by several methods, as summarized in table 2. Due to a comparatively high excitation efficiency and the ease of operation, we use a high-power RF field (35 W) at 150 MHz inductively coupled with an effusing argon beam via a copper coil (10 windings, 30 mm diameter and 100 m length). Atomic beam properties, such as velocity distribution, flux and excitation efficiency, are measured by spectroscopy. We obtain a beam with a mean longitudinal velocity of $\sim 400$ m s⁻¹ with a maximum intensity of $\sim 4 \times 10^{14}$ atoms s⁻¹ sr⁻¹ at a total atom throughput of $\sim 1.5 \times 10^{18}$ atoms s⁻¹ sr⁻¹, which corresponds to an excitation efficiency of $\sim 2 \times 10^{-4}$.

The intensity of the metastable argon beam was found to increase with pressure due to a higher throughput of the atoms. Above a certain pressure, de-excitations caused by collisions with the background gas become important. This loss process occurs when the mean free path $\lambda$ becomes comparable with the dimensions of the vacuum chamber. Accounting for these losses, the metastable beam intensity $I^*$ at a distance $z$ from the source chamber can be written as

$$I^*(z, p) = I(p) \eta_{exc}(p) e^{-z/\lambda(p)},$$

where $I$ denotes the total intensity and $\eta_{exc}$ the excitation efficiency. The latter is derived from the metastable beam intensity measured right behind the source exit (see figure 5) and the total atom throughput given by the pumping speed and the pressure. This pressure dependence of the metastable beam intensity agrees with the experimental data in figure 5, where the metastable beam intensity right behind and at 40 cm from the source exit have been measured as a function of pressure. This result implies that the beam intensity can be enhanced by reducing the size of the high-pressure region.

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5 These parameters have been measured for a source tube of 15 mm diameter and 15 mm aperture, different from the one used in figure 5.
3.2. Collimation

The flux of metastable atoms into the MOT can be greatly enhanced by transverse Doppler cooling of the atomic beam. In order to capture a wide velocity class and to compensate for the changing Doppler shift during the cooling process, we employ a collimation scheme with tilted mirrors [19, 23, 24]. The optimal design parameters have been found through numerical simulations. Representative trajectories together with the corresponding velocities are depicted in figure 6.

The overall performance depends critically on different parameters, such as the laser entry angle $\beta$ and the mirror angle $\theta$ (see figure 7). The simulation reveals that geometrical constraints on the beam propagating through the mirror configuration limit the maximal performance of the collimator. Furthermore, the two angles ($\beta$ and $\theta$) are connected by the condition that the beam is not retroreflected by the last reflection. It is important to note that a change in the mirror angle by only 200 $\mu$rad leads to a drop in the flux enhancement by a factor of 2 (figure 7). Thus, high precision is required for the alignment of the mirror angle.

From measurements of the metastable beam intensity, the power dependence of the flux enhancement at the position of the MOT due to the collimation is calculated (figure 8). This yields a maximum flux enhancement of $\sim 60$ for the available power of $\sim 100$ mW. A further increase is expected to take place by employing higher laser power to approach the maximum of $\sim 90$ predicted by the simulation.
Figure 6. Results from a numerical simulation of the collimation. Simulated trajectories during the collimation process and the final atom beam profile are depicted on the top. The corresponding compression in velocity space together with the final transverse velocity profile is shown below. Only those atoms with a transverse velocity below $\sim 55 \, \text{m} \, \text{s}^{-1}$ can be effectively collimated.

Figure 7. Simulated systematics of the collimation performance for our experimentally available laser power of 200 mW. The sketch of the collimation geometry (left) illustrates the relevant parameters, namely the laser entry angle $\beta$ and the mirror angle $\theta$. The simulation reveals that for a given $\beta$ there is a certain optimum for $\theta$, which coincides with the angle at which the laser beam starts to be retroreflected at the end of the collimation mirror (left plot). The neighbouring graph confirms that this is the case for arbitrary $\beta$ (deviations are due to discretization). Plotting the corresponding maximum flux enhancements (right plot) further reveals an optimum for $\beta$, which coincides with the angle at which the laser beam starts being clipped by the mirror.
Figure 8. Flux enhancement at the position of the MOT due to the collimation versus the total laser beam power. The measured and simulated values agree well in showing a saturating behaviour converging towards a flux enhancement of $\sim 90$.

4. Single atom detection

Even though single-atom detection using fluorescence was realized several years ago [25, 26], setting it up with argon is an important step for the Ar-ATTA apparatus. Our MOT and the fluorescence detection are described in detail in [27]. The following results were all obtained with $^{40}$Ar in order to optimize the detection technique. Fluorescence photons that are scattered from the MOT beams by the atoms at the centre of the trap are coupled into a multimode optical fibre by an objective consisting of two achromatic lenses (see figure 4). This fibre serves as a spatial filter because of its core size of $62.5 \, \mu m$ and its acceptance angle; thus it causes efficient stray light suppression. It guides the photons to an APD integrated in a single-photon counting module (Perkin-Elmer).

For the case of a large number of atoms, we measured the MOT size with the help of a CCD camera and found it to be of the order of the fibre core, $z_{rms} \sim 30–160 \, \mu m$. We define the detectability as the ratio between the calculated photon scattering rate $\gamma_p$ and the cross section $z_{rms}^2$ of the MOT:

$$D = \frac{\gamma_p}{z_{rms}^2}. \quad (3)$$

The dependence on the MOT cross section results from the limitation by the fibre core. $z_{rms}^2$ can be calculated by using a simple model, as in [11]. The motion of the atoms is described by a damped oscillation and the temperature is derived by a random walk approach. This results in

$$D \propto \frac{\gamma_p}{T/\beta} \propto \frac{\tilde{\delta}^2 \cdot s^2}{(1 + s + 4\tilde{\delta}^2)^4}, \quad (4)$$

where $T$ is the temperature of the atoms, $\beta$ the damping coefficient, $\tilde{\delta}$ the laser detuning in units of the natural linewidth and $s$ the saturation. Equation (4) reproduces the main observations.
Figure 9. The atom detectability for three representative detunings. The data are derived from MOT size measurements and the calculated photon scattering rate. The solid curves are derived from theory and fitted to reproduce the functional behaviour of the experimental results. The predicted maximum is calculated from the theory.

 qualitatively. The calculated maximum detectability agrees with the measured value within the error bars. Since the model only involves Doppler cooling, we empirically introduce an effective saturation that reflects sub-Doppler mechanisms. Effective $s$ is extracted by fitting equation (4) via the experimental data. It is by a factor of three larger for small $\delta$ compared with the value for maximum laser detuning. The result is shown in figure 9 and explained as follows.

For $s \ll 1$, a $s^2$-dominated behaviour is predicted for $D$ but cannot be resolved experimentally. The corresponding temperature of the atoms is close to its minimum and the strength of the damping is governed by the low scattering rate. For larger $s$ the increase in scattering rate exceeds the rise in temperature, resulting in larger damping and a small MOT. In the case of high saturation, the curve follows $1/s^2$. The scattering rate saturates and the rise in temperature leads to a larger MOT. The optimum parameters for single atom detection in our system are derived from the measured values to $s \sim 1$ and 9 MHz detuning.

These optimum parameters for single-atom detection are very different from those for optimum capture efficiency. In order to optimize MOT loading, laser detuning as well as saturation ($s$) should be increased. This leads to large capture regions both in real and momentum space. Thus, in the final experiment the MOT parameters will be switched between two sets of parameters: one for loading and the other for fluorescence detection [6].

The expected photon count rate is calculated for the optimum parameters via the well-known scattering rate formula (e.g. (2.26) in [11]). In order to obtain the correct scattering rate, one has to use six times the saturation per MOT beam, leading to $\sim 7$ MHz. The fraction of photons detectable by the APD is limited by several factors, namely the solid angle observed ($\sim 0.6\%$), the transmission of the imaging objective ($T = 0.8$), the coupling to the fibre ($\eta \sim 50\%$) and the sensitivity of the APD (0.53 at 812 nm). Altogether, one expects $\sim 8$ kilocounts per second (kcps) and atom. Figure 10 shows the pulse count rate of the digital
counter as a function of time. The levels indicated have equal spacing of 7.5 kcps, which agrees well with the expected count rate.

The right-hand histogram proves the detection of single atoms, as one can clearly see discrete levels. Comparing the widths of the peaks to their spacings leads to 50 ms detection time, necessary to clearly distinguish one atom and no atom in the MOT. Analysis of the detection peak length allows us to deduce a MOT lifetime of 1 s. As our new setup will have a one order of magnitude lower background pressure ($\lesssim 10^{-8}$ mbar) in the MOT chamber, the lifetime should increase to about 10 s, which means that practically all single atoms will be correctly identified.

The probability of finding a certain number of atoms in the MOT is derived from closer analysis of the histogram. The probability distribution is deduced by fitting Gaussians to the peaks, and it follows a Poissonian distribution, as shown in figure 11. The very good agreement with single-particle statistics again proves the detection of single argon atoms.

5. Summary and outlook

We have discussed several of the essential steps for an $^{39}$Ar-ATTA apparatus. With the hyperfine spectrum now being known, the implementation of atom-optical components for $^{39}$Ar becomes possible. The atomic beam source and a subsequent collimation stage have already been tested for $^{40}$Ar. Possible further improvements include higher RF power for the source discharge, better vacuum in the collimation region and higher laser power for the collimator.

Longitudinal slowing will be achieved in an increasing-field Zeeman slower of 1.8 m length. It slows about 80% of all atoms in the beam down to the capture velocity of the MOT. Single-atom detection in a MOT has been realized for $^{40}$Ar and shows agreement with theory. The MOT will be improved regarding capture region, stray light suppression and background pressure, yielding a detection efficiency for atoms in the MOT near unity.
Figure 11. The probability for different numbers of atoms in the MOT as fitted to the histogram above. The bars are the integrated histogram peaks with their $1\sigma$ confidence intervals. The dashed lines are the probabilities for a Poissonian distribution with a mean value of 1.36. The dashed line for $N = 2$ is not visible as it coincides with the upper bound.

From the measured efficiencies of our components, we can derive the expected $^{39}$Ar count rate for an atmospheric argon sample as follows:

$$\dot{N}_{\text{det}} = I_{\text{max}}^* \cdot R \cdot \eta_{\text{coll}} \cdot \eta_{\text{ZS}} \cdot \eta_{\text{MOT}} \cdot \eta_{\text{HFS}} \cdot \Delta \Omega_{\text{MOT}}$$

$$= 4 \times 10^{14} \text{ atoms s}^{-1} \text{ sr}^{-1} \times 8.5 \times 10^{-16} \times 90 \times 0.8 \times 0.5 \times \frac{2}{5} \times \pi \times 10^{-4} \text{ sr}$$

$$\approx 6 \text{ atoms h}^{-1},$$

(5)

where $I_{\text{max}}^*$ is the metastable beam intensity, $R$ the isotopic ratio in a recent sample, $\eta_{\text{coll}}$ the collimation enhancement, $\eta_{\text{ZS}}$ the Zeeman slower efficiency, $\eta_{\text{MOT}}$ the estimated MOT capture efficiency, $\eta_{\text{HFS}}$ the reduction factor due to hyperfine ground state splitting to five levels and only repumping from one and $\Delta \Omega_{\text{MOT}}$ the solid angle covered by the MOT. Given the resulting count rate of six atoms per hour, we will need around 1 day for 10% accuracy on the ratio $^{39}$Ar/$^{40}$Ar, as 100 atoms have to be detected in this case.

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