

A new normalization method for electron collision cross sections measured using skimmed supersonic jet beams

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Abstract

Crossed electron-molecular beam experiments featuring skimmed nozzle beams may be used to study electron interactions with species such as radicals, excited molecules and biomolecules. A new technique for placing measured cross sections on an absolute scale is reported, as the traditional relative flow method used for effusive molecular beams does not apply to skimmed supersonic jet beams. Absolute cross sections for Ar and CF₄, using this new procedure, are measured and compared to previous effusive beam results as a proof of concept.

Keywords: absolute differential cross sections, supersonic expansion, skimmed supersonic relative density method

1. Introduction

Crossed beam experiments are often used to study electron interactions with atoms and molecules. In such experiments, scattering cross sections are determined by crossing a collimated target beam with an electron beam of known energy, and then measuring the energy and angular distributions of the scattered electrons. Collimated target beams are typically formed as an effusive flow emerging from a single capillary, or multi-capillary, array. Absolute values for the measured cross sections are set subsequently using the well-established relative flow method (RFM) [1].

The effusive-beam RFM has been successfully used to determine absolute differential cross sections (DCS) for a range of neutral atoms and molecules [2]. However, there remain targets of interest for which absolute cross sections are difficult to obtain by means of the RFM. For example, effusive beam methods are not well suited for production of target beams of short-lived species such as those in specific excited states, radicals or biomolecules with low vapour pressures such as DNA bases for example. This is no moot point, as electron scattering cross sections for such targets are required for

modelling processes in research areas including laser and gas discharges, plasma etching of semiconductors, atmospheric processes, and medicinal/biological processes [3].

An alternative method for producing target beams is through the use of a skimmed supersonic expansion (SSE). A SSE involves expansion of the species of interest through a nozzle, and then collimation of the jet into a beam by placing a skimmer(s) downstream of the nozzle. Crossed beam measurements on certain targets may benefit from the use of SSE beams. Specifically, cooling during the expansion produces target beams in which molecules are predominately in the lowest rovibrational state. Optical pumping by a laser might then be used to produce beams with high concentrations of state-specific excited species [3]. In addition, beams containing radicals can be formed by either photolysis or pyrolysis of a parent species in the vicinity of the nozzle, with thermalization of the radical products achieved through collisional cooling in the expansion [4]. Another application might be that biomolecules can be seeded in a carrier gas following laser ablation of an appropriate substrate [5]. Furthermore, beams of polyatomic molecules with low vibrational or rotational temperature can be produced by SSE,

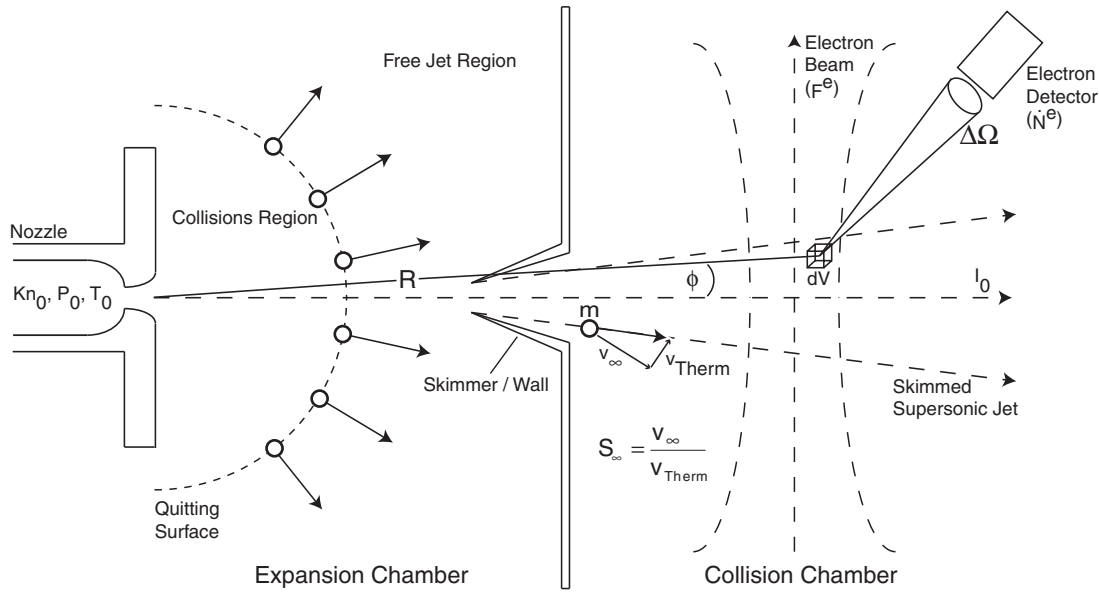


Figure 1. Schematic diagram for the scattering geometry with a skimmed supersonic expansion.

providing a means to test the interaction potentials utilized in theoretical calculations of electron collision cross sections, without the need to resort to comparison with experiments that inherently include the effects of nuclear degrees of freedom. Finally higher centreline intensities are produced by SSE when compared to effusive gas flows, which employ lower stagnation pressures. Hence there are many cases for which a SSE has inherent advantages over the traditional effusive flow approach in crossed beam studies.

To develop a normalization method to obtain absolute DCSs from scattering experiments incorporating SSE beams, the dynamics of an expanding jet beam and any effects of the skimmer/wall on the flow must be understood. Studies of skimmed supersonic gas jets [6–9] yield a description sufficient to determine a normalization method pertinent to SSE, which is effectively equivalent to the RFM. In this paper, a technique to normalize absolute elastic differential cross sections for target beams produced by a SSE is reported. This technique will be referred to as the skimmed supersonic relative density method (SSRDM). In section 2, the scattering geometry in a supersonic expansion is described, and the general form of the SSRDM is presented. In section 3, the experiment used for the proof-of-concept SSRDM measurements reported here is briefly summarized. In sections 4 and 5, differential cross section results from specific applications of the SSRDM technique to molecular and atomic targets are presented. Finally, some conclusions from the present study are drawn in section 6.

2. Skimmed supersonic relative density method

The scattering geometry with a skimmed supersonic expansion is illustrated in figure 1. The absolute cross section (σ) for any scattering process is related to the count rate of the scattered particles (\dot{N}^e) by

$$\dot{N}^e = F^e \eta \sigma \int_{V,E} \rho \Delta\Omega dV dE \quad (2.1)$$

where F^e is the flux of incident electrons, η is the detector efficiency function, ρ is the target density at the scattering centre, $\Delta\Omega$ is the solid angle subtended by the detector, V is the volume of the intersection of the electron and molecular beams, and dE is the range of electron energies. An explicit characterization of all the terms in (2.1) is non-trivial. Absolute normalization is usually achieved by comparing scattering intensities from the target to a reference species with a known DCS [1] e.g. helium. This comparison, in practice, removes the need to explicitly determine the interacting beam fluxes and detector efficiency function. According to (2.1) the unknown (U) DCS of the target is expressed in terms of the reference (R) DCS by

$$DCS_U = DCS_R \frac{\dot{N}_U^e \int \rho_R \Delta\Omega dV dE}{\dot{N}_R^e \int \rho_U \Delta\Omega dV dE} \quad (2.2)$$

The beam density at the intersection of the electron beam and the target molecular beam is given by

$$\rho = \frac{I(\phi)}{v_\infty R^2} \quad (2.3)$$

where $I(\phi)$ is the target beam intensity, v_∞ is the target beam terminal velocity and R is the distance to the gas source. The terminal velocity of ideal gas molecules in a SSE is given by

$$v_\infty = \sqrt{\frac{2k_B T_0}{m} \left(\frac{\gamma}{\gamma - 1} \right)} \quad (2.4)$$

where γ is the adiabatic constant, T_0 is the stagnation temperature, m is the molecular mass, and k_B is Boltzmann's constant. The intensity of the gas beam is expressed as

$$I(\phi) = I_0 J(\phi) \quad (2.5)$$

where I_0 is the centreline beam intensity, and $J(\phi)$ is a geometric function describing the axial intensity distribution. The geometric function for a skimmed nozzle beam [6] is written as

$$J(\phi) = \frac{1}{2\pi} \int_0^{2\pi} [\exp(-S_\infty^2 \sin^2(\phi_1))]_{\phi_{1\max}(\theta,\phi)}^{\phi_{1\min}(\theta,\phi)} d\theta \quad (2.6)$$

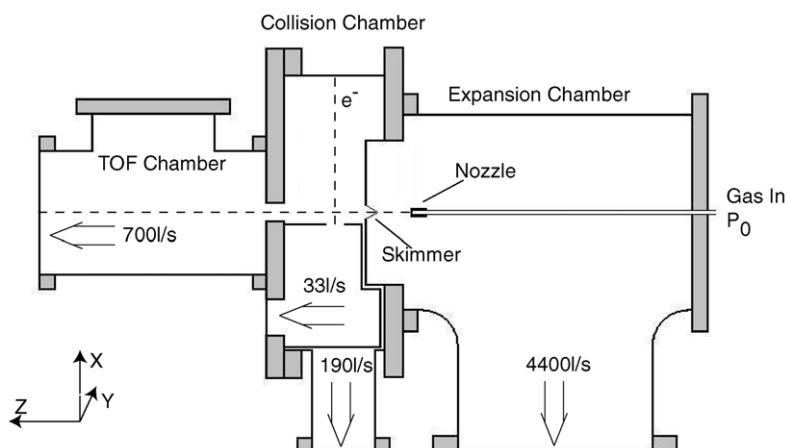


Figure 2. Schematic diagram of the present experimental configuration. The pumping speeds for each of the differentially pumped chambers are shown.

where θ is an angle of rotation about the jet axis, and ϕ is an angle between the normal to the centre of the nozzle and a ray from that point to an off-centreline point in the scattering region. Evaluation of (2.6) is difficult because $\phi_{1\max}$ and $\phi_{1\min}$ are non-trivial functions of ϕ . However, for a molecular beam in free flow at the skimmer, $\phi_{1\max}$ and $\phi_{1\min}$ are determined by the terminal speed ratio (S_∞) and the relevant experimental geometry (see section 4 of [6]). Hence, the only species-dependent term in (2.6) is the terminal speed ratio, i.e. the ratio of translational speed to thermal speed (v_{Therm}) for those molecules expanded beyond the quitting surface [4].

The location of the quitting surface (x_q), at which free molecular flow is achieved, is given as [8]

$$x_q \approx d \left(\frac{S_\infty}{C_1} \sqrt{\frac{2}{\gamma}} \right)^{\frac{1}{\gamma-1}} \quad (2.7)$$

with d the nozzle diameter and C_1 given in table 2. Matching the geometric distribution functions for the unknown beam and the reference beam removes the need for explicit evaluation of the integrals in (2.2). According to (2.6), this condition is achieved by matching the terminal speed ratio of both beams, whilst ensuring free molecular flow exists at the entrance orifice of the skimmer. With these requirements met, the DCS of the unknown molecule is now given as

$$\text{DCS}_U = \text{DCS}_R \left(\frac{I_0}{\dot{N}^e v_\infty} \right)_R \left(\frac{\dot{N}^e v_\infty}{I_0} \right)_U. \quad (2.8)$$

To evaluate (2.8) the terminal speed ratio and centreline intensity of both the target and reference beams at the scattering centre must be determined, with two methods for evaluating these quantities being given later in sections 4 and 5.

The potential for the formation of clusters in a supersonic jet is an important consideration. Dimers or higher order clusters may be formed in the beam if a sufficiently high stagnation pressure behind the nozzle (P_0) is employed. Significant cluster formation will perturb the expansion theory presented here and in sections 4 and 5 by increasing v_{therm} due to the release of energy in forming a van der Waal's bond [7]. The clusters may also be considered an undesired contaminant species in the beam. For a monoatomic gas, dimer concentration exceeding approximately 1% is avoided if the

stagnation conditions are such that the parameter D^* is less than 0.1 [8]. D^* is given as

$$D^* = \frac{P_0 \sigma^3}{\epsilon} \left(\frac{d}{\sigma} \right)^{0.4} \left(\frac{\epsilon}{T_0 k_B} \right)^{2.4} \quad (2.9)$$

where σ and ϵ are the standard parameters of a Lennard–Jones potential, i.e. the finite distance of zero intermolecular force and the depth of the intermolecular potential well respectively. Equation (2.9) is taken as an upper bound to the value of D^* when applied to a polyatomic expansion as the internal relaxations in a molecule release heat into the expansion, which reduces clustering.

A common practice for forming SSE jets is to dilute the gas under study with a lighter gas (e.g. helium or argon). By ‘seeding’ the heavier target gas into a carrier, the target is accelerated to a higher velocity (and consequently cooled to a lower temperature) than would have been achieved in a pure expansion of the target, under the same stagnation conditions. However, [10] notes that the increased cooling achieved in a seeded SSE also increases cluster concentration in the beam. Therefore, as a further precaution to avoiding clustering, carrier gases are not utilized in the experiments reported in sections 4 and 5.

3. Experiment

Only a brief description of the apparatus used to measure the absolute differential cross sections is given here, as full details will be reported elsewhere [11]. The apparatus (figure 2) is a crossed-beam electron scattering experiment comprising three differentially-pumped chambers. The expansion chamber houses the nozzle, the pulsed solenoid valve and the skimmer. The nozzle diameter is 0.8 mm, and the skimmer orifice is 1 mm in diameter. Beams containing molecular radicals are generated by laser photolysis with the addition of a PTFE thermalization adapter [4]. This adapter, manufactured from solid PTFE and mounted to the nozzle flange, provides a region of high collision rate to thermalize the radical daughters prior to expansion. The nozzle-skimmer separation is variable, but is typically set to 50 mm. The valve is pulsed at 10 Hz and individual gas pulses have 3–5 ms duration. Gas is introduced

to the expansion chamber at stagnation pressures in the range of 50–500 mbar.

The collision chamber houses a differentially pumped hemispherical electron spectrometer which produces a collimated electron beam of well-defined energy in the range 0.5–50 eV and with low-energy spread. The gas pulse enters the collision chamber through the skimmer and crosses the electron beam at 90°. The angular distribution of the scattered electrons is measured simultaneously using a fixed-position multi-detector array containing 12 individual detectors. Each detector unit incorporates one channel electron multiplier (CEM), with a retarding field analyser (RFA) mounted before each CEM to prevent detection of inelastically scattered electrons. The combined energy resolution of the spectrometer and RFAs is ~ 1.5 eV [11].

The time-of-flight (TOF) chamber contains a linear, Wiley–McLaren type [12], orthogonal-acceleration time-of-flight mass spectrometer (oa-TOFMS). The oa-TOFMS features 380 mm of field-free flight, two ion acceleration stages producing a second-order space focus, and a 118 nm VUV photoionization source [11]. The purpose of the oa-TOFMS is for the analysis of beams containing multiple species such as molecular radicals and their parent molecules. The oa-TOFMS is therefore not relevant for the present measurements, but is included in this brief description for the sake of completeness.

4. Pressure rise method

The method described in this section for evaluating centreline intensity and speed ratio, is referred to as the pressure-rise skimmed supersonic relative density method (p-SSRDM). In p-SSRDM the centreline intensity of the gas beam downstream of the skimmer is inferred from the pressure rise in the collision chamber once gas is admitted [9]. The centreline intensity is related to the pressure rise (ΔP) by

$$I_0 = \frac{\Delta P S_p R^2}{\alpha k_B T_d} \quad (4.1)$$

where S_p is the pumping speed in the collision chamber for the species under study, α is the cross-sectional area of the molecular beam at the scattering centre, and T_d is the temperature of the collision chamber.

Downstream of the skimmer, the cross-sectional area of the gas beam at the scattering centre is

$$\alpha = \pi \left(\frac{x_d - x_s}{S_\infty} + r_s \right)^2 \quad (4.2)$$

where x_d is the distance to the scattering centre, x_s is the distance to the skimmer (both distances are relative to the nozzle), and r_s is the skimmer radius. The terminal speed ratio for polyatomic molecules is given to within 10% [13] by

$$S_\infty \approx 5.4(P_0 d)^{0.32} \quad (4.3)$$

and with $P_0 d$ in units of Torr cm. The terminal speed ratio for monatomic gases is defined later (see (5.4)). For this work, ΔP was measured with a Bayard–Alpert type ion gauge. To account for the gauge sensitivity to different gas species, the total ionization cross section Q for the species of interest at 150 eV was used [14]. Hence the corrected measured pressure reading (ΔP^*) is given by

$$\Delta P = \Delta P^* \frac{Q_{N_2}}{Q} \quad (4.4)$$

Table 1. Values for Lennard–Jones parameters.

	ϵ/k_B (K)	σ (Å)	Reference
Ar	144.4	3.33	[8]
He	10.9	2.66	[8]
CF ₄	316.9	4.63	[21]

where Q_{N_2} is the total ionization cross section of molecular nitrogen at 150 eV. Nitrogen was used as the reference species to correct the gauge reading as the gauge was originally calibrated for nitrogen. Ionization cross sections for a range of targets, which include some radicals and biomolecules [15, 16], are available in the literature. To further account for species dependence, pumping speeds are scaled as $m^{-1/2}$ [9]. The DCS of the unknown species is then given as

$$DCS_U = DCS_R \left(\frac{\Delta P_d^*}{Q v_\infty m^{1/2} \dot{N}^e} \right)_R \left(\frac{Q v_\infty m^{1/2} \dot{N}^e}{\Delta P_d^*} \right)_U. \quad (4.5)$$

To test the p-SSRDM, the DCS for a polyatomic gas (CF₄) (see figure 3) and for a monatomic gas (Ar) (see figure 4) was measured employing He as the reference gas. The helium cross sections from [17] were used for the normalization. The following ionization cross sections at an energy of 150 eV were also used for the pressure calculation: $Q_{Ar} = 2.68 \text{ \AA}^2$ [18], $Q_{CF_4} = 5.71 \text{ \AA}^2$ [19], $Q_{He} = 0.33 \text{ \AA}^2$ [20]. For the monatomic gas measurement, Ar was introduced at a stagnation pressure of 130 mbar to ensure free molecular flow at the skimmer, with He then introduced at 460 mbar to match the terminal speed ratio of Ar. For the polyatomic experiment, CF₄ was introduced at 120 mbar, and He at 83 mbar. The formation of dimers under these stagnation conditions was checked by evaluating (2.9) for the three gases. The Lennard–Jones parameters used in the calculations are given in table 1. The dimer formation parameter D^* for the three gases was evaluated as $D_{Ar}^* = 3.7 \times 10^{-3}$, $D_{He}^* = 1.95 \times 10^{-4}$ (for He at 460 mbar) and $D_{CF_4}^* = 2.06 \times 10^{-2}$. Dimer formation is therefore considered to be less than 1% in both measurements.

The DCSs determined by the p-SSRDM at a number of energies between 15 eV and 50 eV are compared to the previous RFM measurements for CF₄ [22] and Ar [23, 24]. The present DCSs for CF₄ and Ar are in good agreement with those from the RFM measurements, to within the reported uncertainties at all the energies and angles investigated here. This can be seen in figures 3 and 4. The present DCSs for CF₄ are also seen to be in good accord with the theoretical calculation of [25].

For all the p-SSRDM data presented here, uncertainties are taken as the quadrature sum of the statistical errors (1–2%), uncertainty in the pressure rise (5% each), uncertainty in the speed ratios (10% for polyatomics), uncertainty in the ionization cross sections (2–4%) and the uncertainty of the He DCS (7%). The total uncertainty in the present DCSs is therefore in the range of 25–30%, compared with the 10–20% range reported on the RFM measurements. The main limitations in reducing the absolute uncertainty of the current p-SSRDM DCS were the small pressure rises (ΔP) measured in the collision chamber and the uncertainty in the calculated speed ratio. In the present configuration, pressure rises in the collision chamber were as low as 20% with a pressure gauge precision of about 2% of total gas pressure. A system

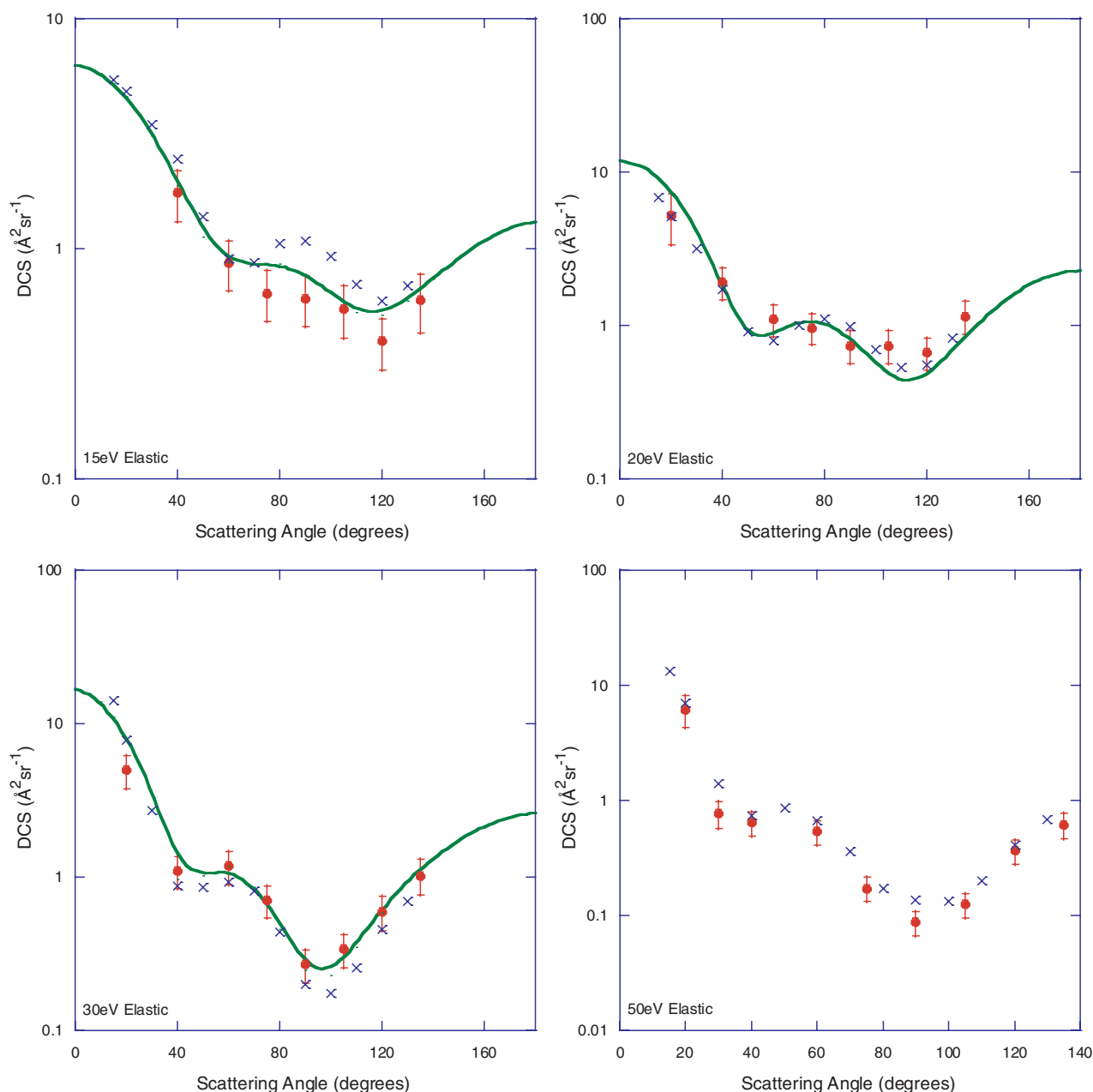


Figure 3. Absolute elastic DCS ($\text{\AA}^2 \text{sr}^{-1}$) for CF_4 at various energies. Present results obtained by the p-SSRDM are shown (\bullet). The effusive beam experiments of Boesten *et al* [22] (\times) and the multichannel Schwinger calculation of Varella *et al* [25] (—) are also shown.

with a continuous gas source, and/or a third ‘dump’ chamber with a smaller volume to achieve a more significant pressure rise could conceivably achieve smaller uncertainties than those reported here. Alternatively, a more detailed study into the speed ratios of polyatomic molecules would reduce the uncertainty introduced using the approximation for the speed ratio given by equation (4.3).

5. The transmission method

As an alternative approach, the target density at the scattering centre for a skimmed supersonic molecular beam can be determined using a method which is entirely based on transmission theory [6–8]. This approach is referred to here as

the transmission skimmed supersonic relative density method (t-SSRDM). Cross sections with less uncertainty than those from the p-SSRDM can be determined using the t-SSRDM, as the uncertainties on the measured parameters (ΔP , S_{∞}^* , Q) are not introduced into this new normalization method. However, the theoretical description for the interaction of the skimmer-wall system with a supersonic jet is only accurate for monatomic gases [6–8].

In the absence of a skimmer, the ideal centreline intensity (\mathcal{I}_0) at the scattering region downstream from the nozzle [7, 8] is defined as

$$\mathcal{I}_0 = \frac{\kappa \dot{N}}{\pi} = \frac{\kappa F(\gamma) n_0 d^2}{4} \sqrt{\frac{2k_B T_0}{m}} \quad (5.1)$$

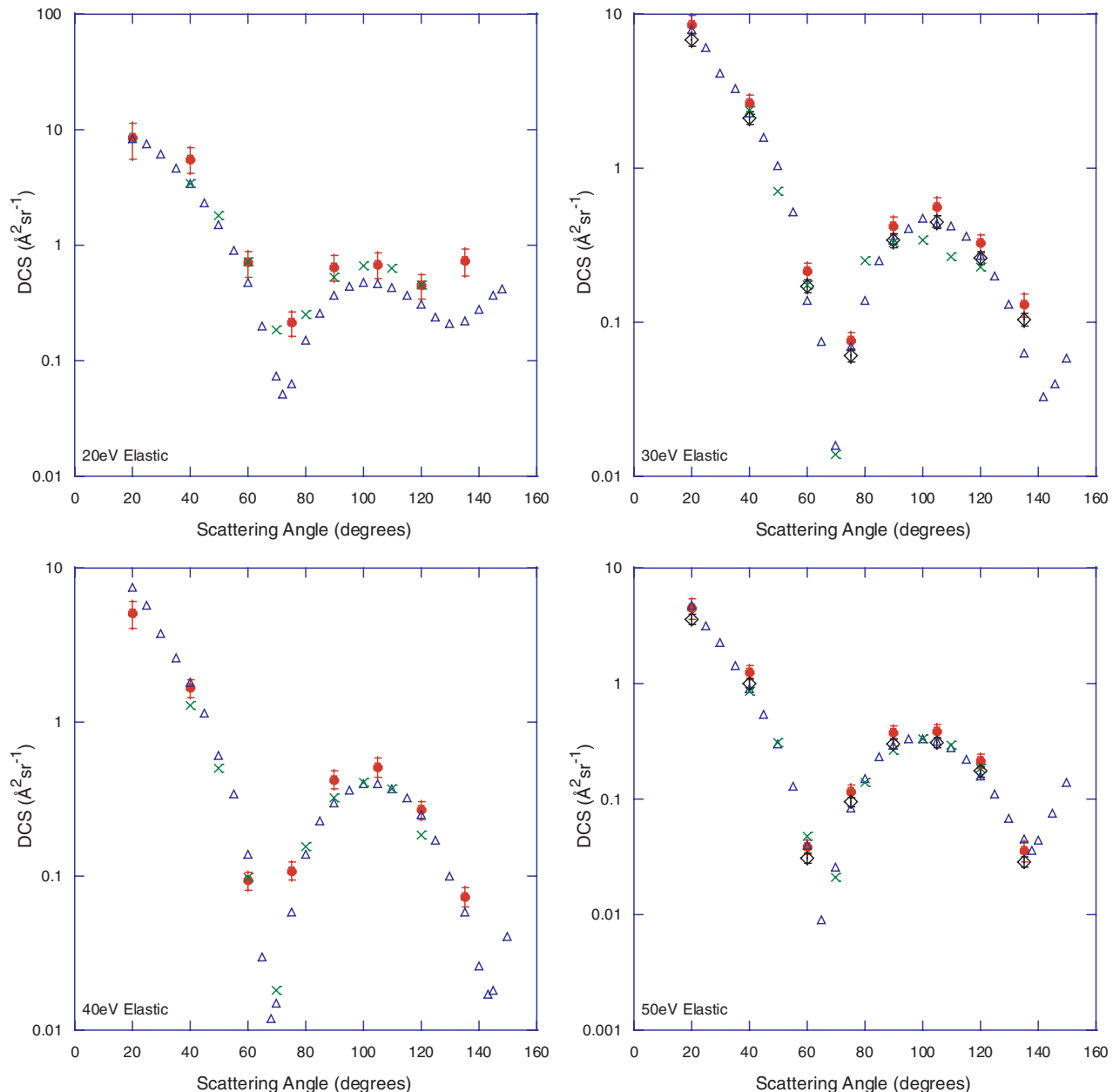


Figure 4. Absolute elastic DCS ($\text{\AA}^2 \text{sr}^{-1}$) for argon at various energies. Present results (\bullet) obtained by the p-SSRDM are shown, as well as by t-SSRDM at 30 and 50 eV (\diamond). The effusive beam measurements of Cvejanovic *et al* [23] (\times) and Panajotovic *et al* [24] (\triangle) are also shown.

where κ is the peaking factor, \dot{N} is the flow rate, n_0 is the stagnation density and d is the nozzle diameter, with

$$F(\gamma) = \left(\frac{\gamma}{\gamma+1} \right)^{1/2} \left(\frac{2}{\gamma+1} \right)^{1/(\gamma-1)}. \quad (5.2)$$

The values of γ and κ for different gases are given in table 2. With a skimmer in place, the centreline intensity of the gas beam in the scattering centre is less than the ideal intensity due to interference resulting from the presence of the skimmer-wall system [8]. There are three mechanisms by which the centreline intensity is reduced from that of the ideal. First, molecules not in the centreline of the beam, but with sufficient thermal velocity to reach the scattering centre, are blocked. Second, jet molecules scattered by the skimmer and the wall

Table 2. Terminal speed ratio constants. Alternate experimental values are indicated in parentheses. This table has been reproduced from Miller [8].

γ	κ	A	B	C_1
5/3 (monatomic)	1.98 (2.0)	0.527 (0.778)	0.545 (0.495)	3.232
7/5 (diatomic)	1.38 (1.47)	0.783	0.353	3.606
9/7 (polyatomic)	1.11 (1.18)	1.022	2.610	3.971

may reflect back into the path of the centreline beam resulting in attenuation. Finally, scattering by background gases reduces centreline intensity further below the ideal.

The centreline intensity downstream of the skimmer, relative to the ideal intensity (neglecting background

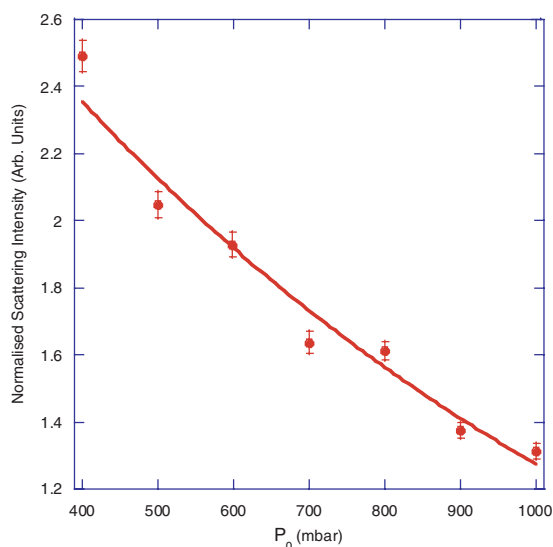


Figure 5. A typical result for Beers attenuation, shown here in 105° scattering for argon.

scattering) [6], is given by

$$I_0 \approx I_0 \left(1 - \exp \left[- \left(\frac{S_\infty r_s x_d}{x_q (x_d - x_s)} \right)^2 \right] \right). \quad (5.3)$$

Equation (5.3) assumes hypersonic flow ($S_\infty > 5$) and $x_q < x_s$. The terminal speed ratio for a monatomic gas [8] is given as

$$S_\infty = A [Kn_0]^B \quad (5.4)$$

where Kn_0 is the source Knudsen number, and the parameters A and B are given in table 2. Equation (5.4) is in principle extendable to polyatomic targets, but the results should be considered to be less reliable [8] as the release of energy following internal relaxations in the molecules perturbs the ideal expansion.

The effect of background scattering on centreline intensity is accounted for by including a Beer's attenuation term, $I_0/I_0 = \exp(-bP_0)$ [8]. By varying the driving pressure and normalizing the scattering intensity by the skimmer attenuation term (equation (5.3)), Beer's attenuation constant b was measured (see figure 5). Attenuation constants for both the unknown, b_U , and reference, b_R , gases were determined and then included in the t-SSRDM equation. The final form of the t-SSRDM DCS normalization is then

$$\text{DCS}_U = \text{DCS}_R \frac{\dot{N}_U^e [v_\infty m^{1/2}]_U [P_0 \exp(-bP_0)]_R}{\dot{N}_R^e [v_\infty m^{1/2}]_R [P_0 \exp(-bP_0)]_U}. \quad (5.5)$$

To test the t-SSRDM approach, the absolute elastic DCSs for Ar were measured and then cross checked against our corresponding p-SSRDM results (figure 4), and the results of other effusive beam studies [23, 24]. The monatomic DCSs produced by t-SSRDM and by p-SSRDM agree to within the combined uncertainties across the entire angular range. Similarly good agreement was seen when our t-SSRDM cross sections were compared with the results of the earlier studies [23, 24].

Uncertainties in the t-SSRDM method include the quadrature sum of statistical errors (1–2%), uncertainty in the

Beer's constant (<1%) and the uncertainty in the helium DCS (7%). The total uncertainty in the measured DCS is therefore about 10%, which is consistent with the uncertainties reported from the RFM measurements.

6. Conclusions

Two methods for setting absolute scales for elastic differential cross section measurements, made in crossed-beam experiments using skimmed supersonic nozzle sources, have been described. The p-SSRDM approach relates the target density at the interaction region to the pressure rise in the collision chamber once gas is admitted. DCSs for Ar and CF_4 gases were measured using p-SSRDM and shown to be consistent with previous RFM data. The total uncertainty in the cross sections was 1.5–2.5 times higher than the uncertainties reported in the literature RFM measurements. This fact is due to the propagation of measurement errors on the normalization parameters. However, design of an experiment which could produce reduced p-SSRDM uncertainties compared to those reported here is feasible.

The t-SSRDM method uses transmission theory to calculate centreline intensity at the scattering centre. DCSs for Ar were measured and shown to be consistent with those determined by the p-SSRDM and with the previous measurements. Uncertainties with this approach were seen to be about 50% smaller than those obtained by the p-SSRDM, but the method is currently only applicable to monatomic targets.

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