

Low-Temperature Oxygen Plasma Ashing for Environmental and Occupational Analysis

Asbestos, respirable crystalline silica and coal mineral matter: mechanism, standard methods and test data from the Tergeo plasma asher

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Abstract

Many environmental and occupational measurements look for a small amount of mineral matter inside a much larger amount of organic material: asbestos fibers on a cellulose-ester air filter, quartz on a PVC filter, clay and pyrite grains in coal, fibers in lung tissue. An oxygen plasma removes the organic part as CO, CO₂ and H₂O while the sample stays far below furnace temperatures. That combination is the reason plasma ashing is written into AHERA, ISO 10312, ISO 13794, EPA Method 100.2, NIOSH 7500, 7602 and 7603 and MSHA P-7, and why coal geologists have relied on it since the 1960s. This application note explains how low-temperature oxygen plasma ashing (LTA) works, why temperature decides whether the analyte survives, and how direct, shielded and remote (downstream) exposure change what reaches the sample. We then report tests on Tergeo-series plasma systems. A 47 mm cellulose acetate membrane filter was ashed in about 20 min at 30 W and about 60 min at 15 W (90 sccm O₂), while a thermocouple under the glass dish rose by only 8 °C and 6 °C. A 25 mm filter inside a foil-capped glass vial was removed in 1–2 h at 75 W. Coal powders (0.87–1.10 g) and charcoal ashed at 75 W and 10 sccm O₂ reached their emission end points in 3–6 h, three of the four without stirring, and the coals left 6–14 % residue. In every run that was logged, the plasma emission signal showed the end point without opening the chamber.

Keywords: *low-temperature plasma ashing; plasma asher; LTA; asbestos TEM sample preparation; MCE filter plasma etching; AHERA; ISO 13794; respirable crystalline silica; NIOSH 7500; coal mineral matter; Tergeo*

1 Introduction

Most samples that reach an environmental or industrial-hygiene laboratory consist largely of organic material. An asbestos air sample is a cellulose-ester membrane that outweighs the dust it collected many times over. A vinyl floor tile or roofing felt holds its fibers in asphalt or polymer. A respirable-dust filter for silica analysis is made of PVC, and coal is mostly carbon. Before a microscope, an X-ray diffractometer or an infrared spectrometer can measure the mineral fraction, the organic material has to go, and whatever removes it must not change, lose or add to the particles the analyst is trying to count, identify or weigh.

The regulatory limits are low. OSHA limits airborne asbestos to 0.1 fiber/cm³ as an 8-hour time-weighted average, counting fibers 5 µm or longer with a length-to-width ratio of at least 3:1 [1]. A building material counts as asbestos-containing above 1 % asbestos [2]. After an abatement in a school, the inside air samples must average no more than 70 structures/mm² of filter area by TEM [3]. In March 2024 the EPA issued a rule under TSCA that ends ongoing uses of chrysotile [4]. For respirable crystalline silica, the OSHA permissible exposure limit is 50 µg/m³ [5], and MSHA adopted the same limit for mines in 2024 [6]. At these levels, small losses or gains during preparation show up in the result.

Laboratories have three ways to remove an organic matrix. Wet digestion with acids, bleach or alkali dissolves it. Dry ashing in a muffle furnace burns it, typically at 450–600 °C for filters and bulk materials and

at 750 °C or more for the standard coal ash test [7–9]. Low-temperature plasma ashing oxidizes it with oxygen atoms made in a radio-frequency (RF) discharge, while the sample stays far cooler than a furnace. Gleit and Holland described the technique in 1962 for biological and organic samples [10], and Gluskoter applied it to coal in 1965 [11]. It has since become a standard preparation step in asbestos, silica and coal mineralogy methods.

This note has two parts. Sections 2 to 5 explain where plasma ashing enters each method, how the chemistry works, why the low temperature matters for each analyte, and how the way the sample meets the plasma changes the result. Section 6 reports our own tests on Tergeo-series plasma systems with cellulose acetate membrane filters, filters in vials, coal, charcoal and cellulose wipes, including the sample temperature and the plasma-emission end point. Section 7 turns the results into starting recipes and quality checks.

2 Where plasma ashing fits in environmental and occupational methods

The word "ashing" covers three different jobs, and a recipe that suits one of them is wrong for the other two. In a **surface etch**, the plasma removes only the top 1–2 µm of a collapsed membrane filter so that fibers trapped in the surface are exposed but stay where they landed. In **full ashing**, the whole filter and any organic dust on it are oxidized, and the inorganic residue is redispersed or redeposited for analysis. In **matrix ashing**, grams of organic binder, tissue or coal are removed to concentrate the mineral fraction. Figure 1 maps the three jobs onto the samples and methods covered in this note, and Table 1 lists what each method specifies.

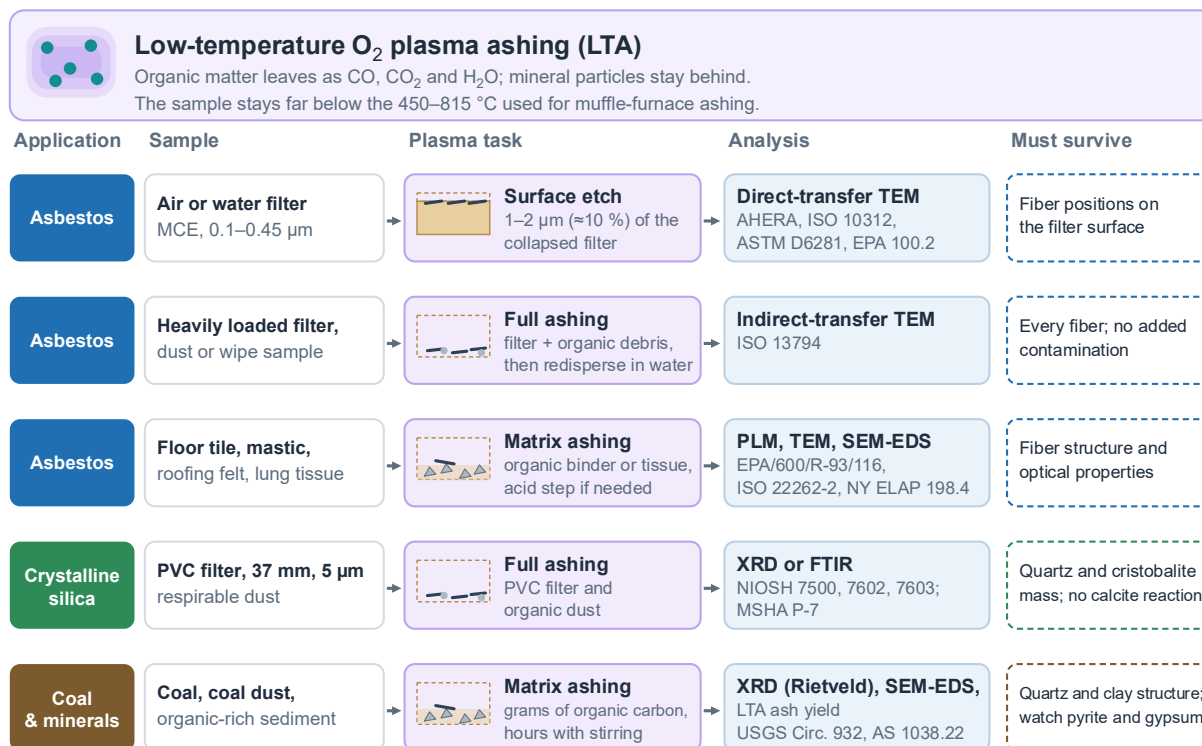


Figure 1. Low-temperature oxygen plasma ashing in environmental and occupational analysis. The same instrument performs three different jobs: a shallow surface etch for direct-transfer TEM, full ashing of a filter for indirect TEM or silica analysis, and matrix ashing of bulk materials, tissue or coal. The right-hand column lists what each preparation must leave unchanged.

Table 1. Standard methods that specify, permit or omit plasma ashing.

Method	Sample and analysis	Plasma step	What the method specifies
AHERA, 40 CFR 763 Subpart E, App. A [3]	Air; MCE $\leq 0.45 \mu\text{m}$; direct-transfer TEM	Surface etch of the collapsed filter (required)	Remove a 1–2 μm ($\approx 10\%$) layer; set the time from the ashing rate of a known weight of collapsed filter; oxygen supply and bleed valve; carryover blank
ISO 10312; ASTM D6281 [12,13]	Ambient and indoor air; MCE or cellulose nitrate; direct-transfer TEM	Surface etch	Etch "to ensure that all particles are exposed"; asher conditions set by calibration (ISO 10312 Annex A)
EPA Method 100.2 [14]	Drinking water; MCE 0.1–0.22 μm ; TEM	Surface etch: required for 0.22 μm , recommended for 0.1 μm	Remove about 10 % of the filter mass
ISO 13794 [15]	Ambient air, heavily loaded filters; indirect-transfer TEM	Full ashing	Ash in an oxygen plasma; disperse residue in water at pH 3–4; refilter on PC $\leq 0.2 \mu\text{m}$ or CE $\leq 0.22 \mu\text{m}$
ISO 14966 [16]	Air; gold-coated PC filter; SEM-EDS	Removal of organic particles	Technically pure oxygen; asher calibration procedure in the standard
NIOSH 7400 and 7402 [17–19]	Air; PCM count and TEM asbestos fraction	None	Filters cleared with acetone or DMF solution; LTA was removed from 7402 because it destroyed organic fibers
EPA/600/R-93/116 [7]	Bulk building materials; PLM, XRD, TEM	Matrix ashing: plasma for small cellulosic samples, or furnace	Furnace 300–500 °C, usually 6 h at 450 °C; chrysotile decomposes above ≈ 500 °C; track mass at each step
NY ELAP 198.4 and 198.6 [20]	NOB bulk (floor tile, mastic, roofing); TEM or PLM	Matrix ashing in a furnace	485 $\pm$ 5 °C to constant mass, then HCl; only TEM can show an NOB sample is non-asbestos
NIOSH 7500 [8]	Respirable dust on PVC; XRD	Full ashing (or furnace, or THF)	LTA in 50-mL beakers; residue in 2-propanol, redeposited on 0.45 μm silver; furnace 2 h at 600 °C (800 °C with graphite)
NIOSH 7602 [21]	Respirable dust on PVC; FTIR (KBr pellet)	Full ashing (or furnace)	LTA in aluminum dishes; furnace 2 h at 600 °C; HCl wash before furnace ashing if calcite >20 %
NIOSH 7603; MSHA P-7 [22,23]	Respirable coal mine dust on PVC; FTIR after redeposition	Full ashing (or furnace for 7603)	LTA 2 h at 300 W and 75 sccm O ₂ (7603); about 60 min (P-7); kaolinite correction after LTA
Coal mineral matter: AS 1038.22; USGS Circ. 932 [24,25]	Coal; LTA yield, XRD, SEM-EDS	Matrix ashing	Low-temperature plasma, typically 100–200 °C; thin layer, periodic stirring, ash to constant mass

CE, cellulose ester; LTA, low-temperature (plasma) ashing; NOB, non-friable organically bound; PC, polycarbonate.

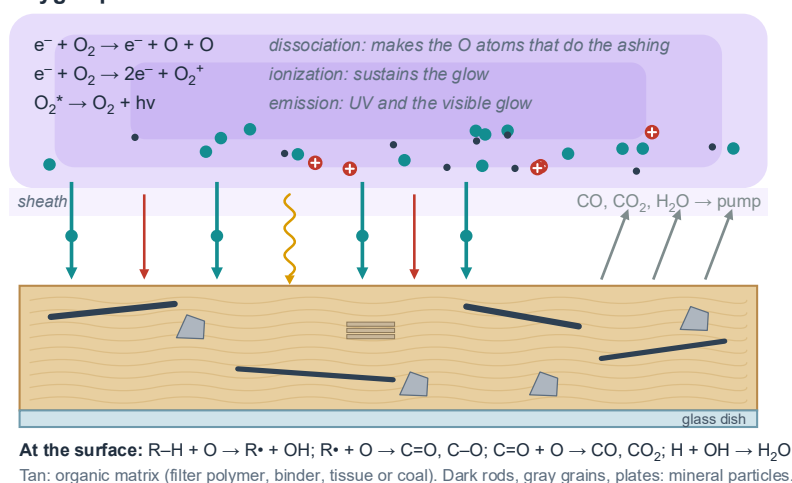
Two widely used asbestos methods have no plasma step at all. NIOSH 7400 counts fibers by phase-contrast microscopy on a filter cleared with acetone or a DMF solution [17]. NIOSH 7402 then uses TEM to find what fraction of those optically counted fibers is asbestos. An earlier revision ashed the filter at low temperature, but that step removed organic fibers such as cellulose and so biased the asbestos fraction upward; it was dropped [18,19]. Plasma ashing should be added to a method only where the method calls for it.

3 How low-temperature oxygen plasma ashing works

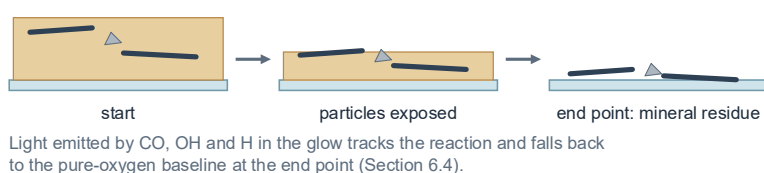
3.1 Reactive species and surface reactions

A low-pressure RF discharge in oxygen, typically at 0.1–1 Torr and 13.56 MHz, accelerates free electrons that then collide with O₂ molecules (Figure 2). Some collisions split the molecule into two oxygen atoms; others ionize it or excite it so that it emits ultraviolet and visible light. The oxygen atoms do most of the ashing. In the polymer-etching literature, atomic oxygen is the species that starts the reaction, while ions and vacuum-UV photons from the glow speed it up by breaking bonds at the surface [26]. Hydrogen is abstracted from C–H bonds, oxygen adds to the carbon radicals left behind, and the chain is cut repeatedly until the fragments leave as CO, CO₂ and H₂O. Organic sulfur and nitrogen leave as their oxides [25]. The vacuum pump removes these products, and what remains on the dish is the inorganic part of the sample.

Oxygen plasma



Progress of ashing



Why the sample stays cool

Energy goes to the electrons
electrons: 1–5 eV (≈10,000–60,000 K)
gas and ions: a few hundred kelvin

electrons

gas, ions, sample

Heat that does reach the sample

- oxidation of the organic matter
- ion and electron recombination
- UV absorbed at the surface
- conduction from warm chamber walls

Measured in Tergeo

(thermocouple under the glass dish)

15 W, 90 sccm O₂: +6 °C

30 W, 90 sccm O₂: +8 °C

75 W, 10 sccm O₂: 27 → 43 °C in 15 min

For comparison

muffle-furnace ashing in standard

methods: 450–600 °C (coal ash: 750–815 °C)

Figure 2. Mechanism of low-temperature oxygen plasma ashing. Electron collisions in the glow make oxygen atoms, ions and UV photons. Oxygen atoms reaching the surface oxidize the organic matrix step by step to volatile CO, CO₂ and H₂O, which are pumped away, and the mineral particles settle onto the dish. The panel on the right summarizes why the sample stays cool and gives the temperatures we measured in Tergeo systems.

Because the reaction happens at the surface, the rate depends on the area exposed and on how easily oxygen atoms reach material below the surface. A thin membrane is attacked from the whole of one face and disappears in minutes. A bed of coal powder ashes from the top down, and the mineral ash that builds up on top slows the attack on the coal beneath it. Miller's account of coal-ashing practice gives the practical consequence: the sample must be spread thinly and stirred at intervals, or the ashing time grows to days [25]. The glow also records the reaction. Emission from excited CO, OH and H appears when organic material is being oxidized and fades when it is gone, which is the basis of optical end-point detection in photoresist stripping [27,28] and of the end-point traces in Section 6.

3.2 Why the sample stays cool, and why that matters

An RF plasma is far from thermal equilibrium. Electron temperatures are 1–5 eV, roughly 10,000–60,000 K, while the neutral gas and the ions in the glow stay at a few hundred kelvin [29]. The chemistry is driven by the electrons, so the oxygen atoms are made without heating the gas, and the sample is heated only by the oxidation itself, by oxygen atoms, ions and electrons recombining on its surface, by absorbed UV and by contact with the chamber walls. How warm the sample gets therefore depends on the RF power, the pressure, how much organic material is burning and how well the sample holder conducts heat away.

Several groups have measured how warm the sample gets. In a nine-laboratory comparison of coal ashing, Miller reported that "the plasma operates at 100–200 °C", with higher RF power giving higher temperatures [25]. Han and co-workers measured chamber temperatures of 88, 121, 149 and 177 °C at 100, 200, 300 and 400 W in a commercial parallel-plate asher, and 123 °C at 75 W in another tabletop unit [30]. Gylseth and co-workers kept lung tissue below 200 °C [31]. In our Tergeo tests (Section 6.3), a thermocouple under the glass dish rose by 6 °C at 15 W and 8 °C at 30 W during membrane-filter runs, and by 16 °C in the first 15 min of a 75 W run on a carbon powder. All of these are well below the 450–815 °C used for furnace ashing.

Figure 3a places these process temperatures next to the temperatures at which the analytes start to change. The thresholds explain most of the method rules in Section 5.

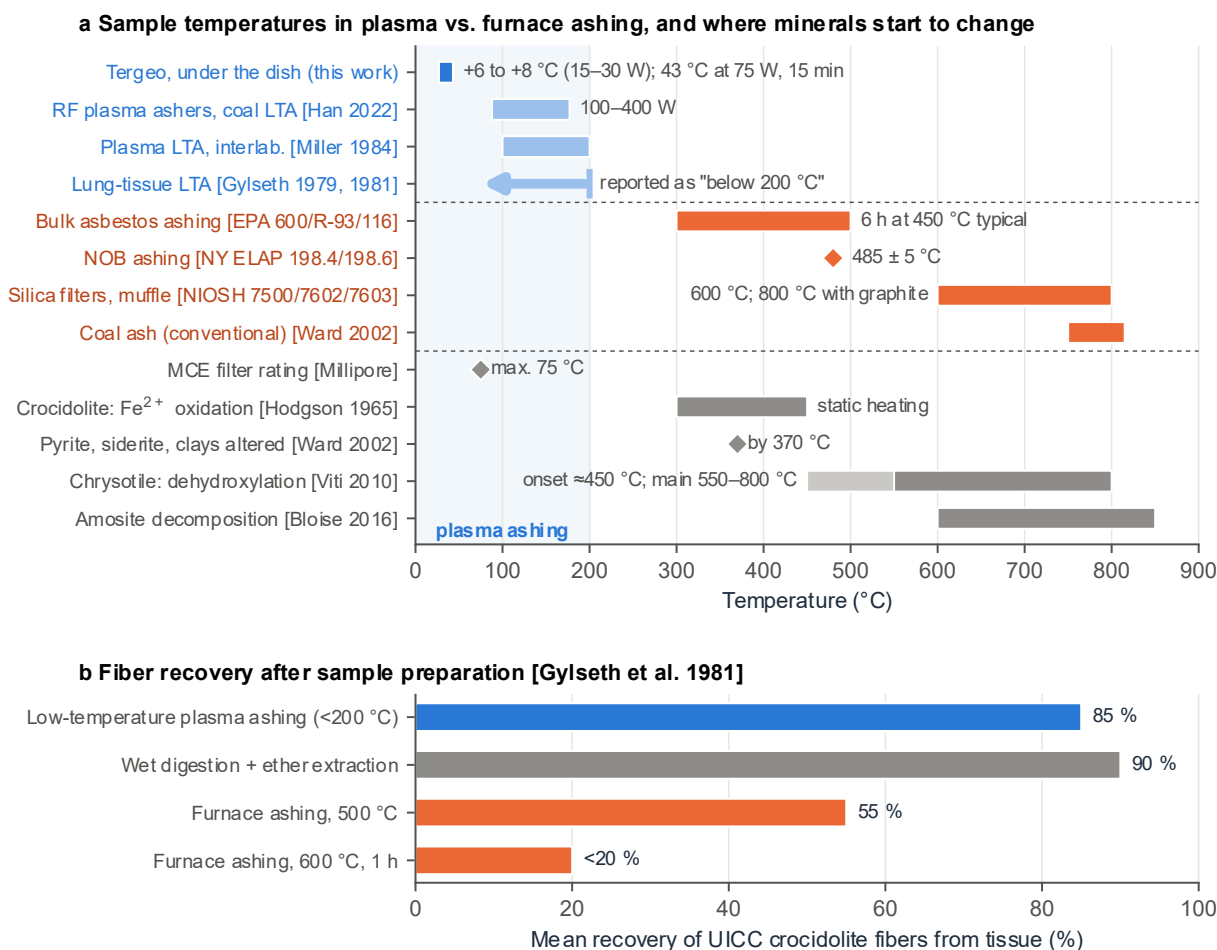


Figure 3. (a) Sample temperatures reported for plasma ashing (blue), temperatures specified for furnace ashing in standard methods (orange), and temperatures at which filters and minerals begin to change (gray). Sources: Tergeo

measurements, this work; Han et al. [30]; Miller [25]; Gylseth et al. [31,32]; EPA/600/R-93/116 [7]; NY ELAP 198.4/198.6 [20]; NIOSH 7500, 7602, 7603 [8,21,22]; Ward [9]; MCE filter rating [33]; Hodgson et al. [34]; Viti [35]; Trittschack and Grob  ty [36]; Bloise et al. [37]. (b) Recovery of UICC crocidolite fibers added to tissue after four preparation methods, from Gylseth et al. [32].

- **Membrane filters.** A mixed cellulose ester (MCE) membrane is rated to 75 °C [33]. A collapsed filter that softens, shrinks or curls during etching moves the particles it is meant to hold in place.
- **Amphibole asbestos.** Crocidolite begins to lose hydrogen and oxidize its iron at 300–450 °C under static heating [34], and heat-treated sodic amphiboles show higher refractive indices and birefringence [38]. NIOSH 9002 warns that heat and acid treatment can change the refractive index and color of asbestos [39], and OSHA cautions that heating for 1–2 h, even below 600 °C, may measurably change optical properties [40]. Optical properties are exactly what PLM uses to identify the mineral.
- **Chrysotile.** Dehydroxylation runs mainly from 550 to 800 °C [35], but in situ Raman work shows a talc-like phase forming at the fiber edges from about 450–460 °C [36]. The EPA bulk method states that chrysotile "will decompose above approximately 500 °C" and caps furnace ashing at 500 °C [7].
- **Coal minerals and silica.** Pyrite, siderite and some clays change irreversibly even at 370 °C [9]. In a muffle furnace, calcite that makes up more than about 20 % of the dust reacts with quartz and lowers the quartz result [21,22].

Gylseth and co-workers added UICC crocidolite to tissue and recovered a mean of 85 % after low-temperature plasma ashing and 90 % after wet digestion with ether extraction, compared with 55 % after furnace ashing at 500 °C and less than 20 % after 1 h at 600 °C (Figure 3b) [32]. Plasma ashing kept nearly all of the recovery of a wet method while leaving a dry residue. A 2025 study of rat lung exposed to Libby amphibole found pitting and gaps along fibers ashed in a muffle furnace at 485 °C and concluded that furnace ashing was unsuitable for mineralogical work on those samples [41].

Low temperature does not make plasma ashing chemically inert, and a careful method accounts for that. Oxygen atoms still oxidize pyrite, gypsum loses water to form bassanite above about 100 °C, and organic sulfur can be fixed as sulfate in coal ash [25]. Han and co-workers found that coal LTA above about 150 °C began to degrade clay mineral structures, while below 80 °C the organic matter was not removed completely [30]. Some trace elements are partly lost: on spiked membrane filters, Imai recovered 55–94 % of arsenic, cadmium and selenium, against more than 95 % for most other elements apart from mercury, bromine and iodine [42]. For mineral and fiber analysis, those effects are small compared with what a furnace does. For volatile trace elements, recovery has to be validated.

3.3 Chamber surfaces and the quartz chamber

Oxygen atoms are lost when they recombine to O₂ on a surface. The fraction of wall collisions that end this way, the recombination coefficient γ , varies by orders of magnitude between materials (Figure 4). Room-temperature values reported for quartz glass lie mostly between 2×10^{-5} and 4×10^{-3} , while stainless steel, copper, silver and nickel lie between about 2×10^{-3} and 0.4 [43]. Aluminum spans the whole range depending on its surface state.

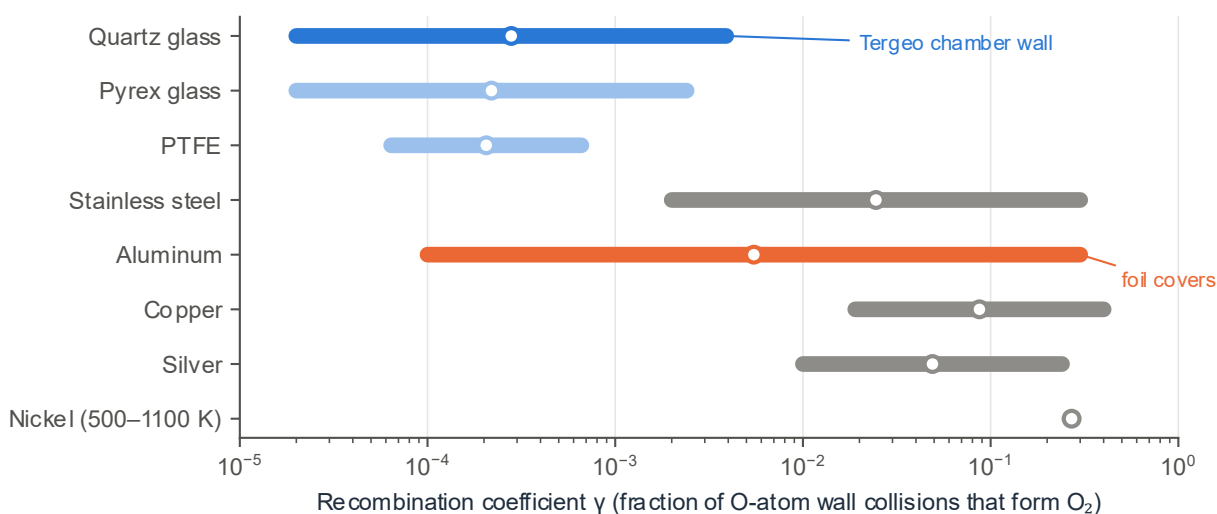


Figure 4. Reported recombination coefficients of oxygen atoms on chamber and sample-holder materials, compiled from the review by Paul et al. [43]. Bars show the range of values reported, mostly near room temperature; circles mark the geometric mean of the range. Quartz, the Tergeo chamber material, consumes far fewer oxygen atoms per wall collision than metals.

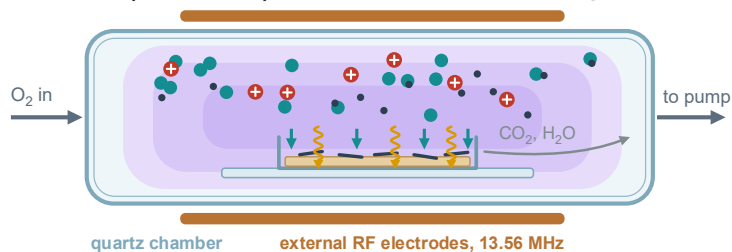
For practical work, a quartz chamber keeps the oxygen-atom density high and puts no metal into contact with the sample, which matters when the residue goes to EDS or trace-element analysis. A metal cover over a sample, such as perforated aluminum foil, consumes part of the oxygen atoms passing through it, which is one reason a foil-capped vial ashes more slowly than an open dish (Section 6.5). And in remote (downstream) operation, where the atoms travel from a separate source to the sample, a low-recombination path is what lets enough of them arrive.

4 Three ways to expose the sample: direct, shielded and remote

How the sample meets the plasma changes which species reach it, how fast it ashes and how warm it gets. Figure 5 compares the three arrangements available on Tergeo systems; pulsed operation can be combined with any of them.

A Direct (immersion) mode

the sample sits inside the glow

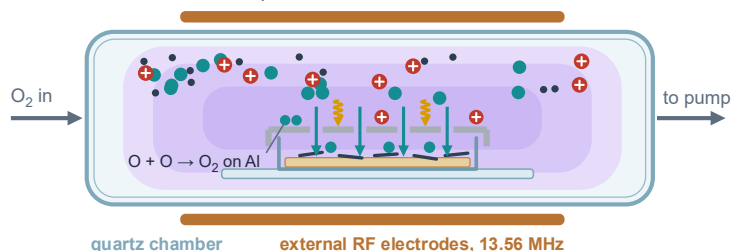


What reaches the sample

- O atoms + ions + electrons + UV
- **Fastest ashing**
complete ashing of filters, coal and organic-bound bulk material
- **Most sample heating of the three**
PIE: +8 °C under the dish at 30 W

B Shielded mode

a perforated cover sits over the dish

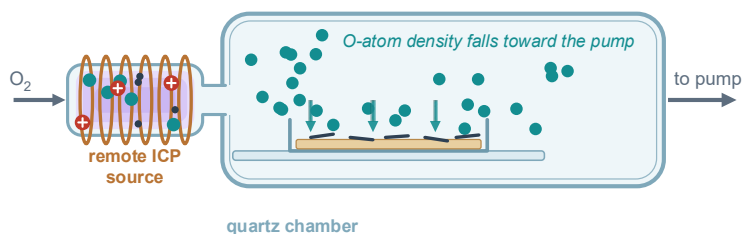


What reaches the sample

- **Mainly O atoms, through the holes**
- **The cover stops most ions and UV**
and keeps light, loose ash in the dish
- **Slower than direct mode**
aluminum recombines part of the O atoms

C Remote (downstream) mode

the plasma is made in a separate source

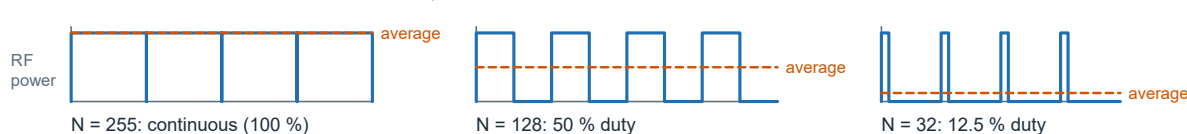


What reaches the sample

- **Long-lived neutrals (O atoms)**
- **No glow, ions or UV at the sample**
gentlest treatment, least heating
- **Slowest; rate depends on position**
optional on Tergeo and Tergeo-Plus

D Pulsed mode

can be combined with A, B or C



Average power = set power × N/255. The duty cycle runs from 100 % down to below 1 %, so a 75 W recipe can be run at a few watts on average for slow, controllable etching or for heat-sensitive samples.

● O atom ⊕ O₂⁺ / O⁺ ion ● electron ⚡ UV photon — mineral particle ■ organic matrix (filter, binder, coal)

Figure 5. Three ways to expose a sample to an oxygen plasma. (A) Direct (immersion) mode: the sample sits in the glow and receives oxygen atoms, ions, electrons and UV. (B) Shielded mode: a perforated cover over the dish blocks most ions and UV, admits oxygen atoms through the holes and keeps loose ash in place, at some cost in rate. (C) Remote (downstream) mode: the plasma is made in a separate inductively coupled source, and only long-lived neutral species reach the sample. (D) Pulsed mode sets the fraction of time the RF power is on, lowering the average power for slow, controllable etching.

Direct (immersion) mode. In Tergeo systems the direct-mode plasma is a capacitively coupled discharge driven through external electrodes around the quartz chamber [44]. The sample sits inside the glow and receives the full flux of oxygen atoms, ions, electrons and UV. This is the fastest way to ash and the right choice for full ashing of filters, coal and organic-bound bulk materials. It also heats the sample most, although in our filter tests the rise under the dish was still under 10 °C (Section 6.3).

Shielded mode. Laboratories often place a perforated cover, usually aluminum foil with pin holes, over a dish or vial. The cover sits in the glow and intercepts most of the ions and UV, while oxygen atoms diffuse through the holes to the sample. It also stops the light, porous ash from being lifted when the chamber is pumped or vented. The cost is speed: the foil recombines part of the oxygen atoms (Figure 4), and the holes limit how many reach the sample. Slow pumping and slow venting, available on Tergeo systems, are another way to protect a fragile residue, without a cover in the way.

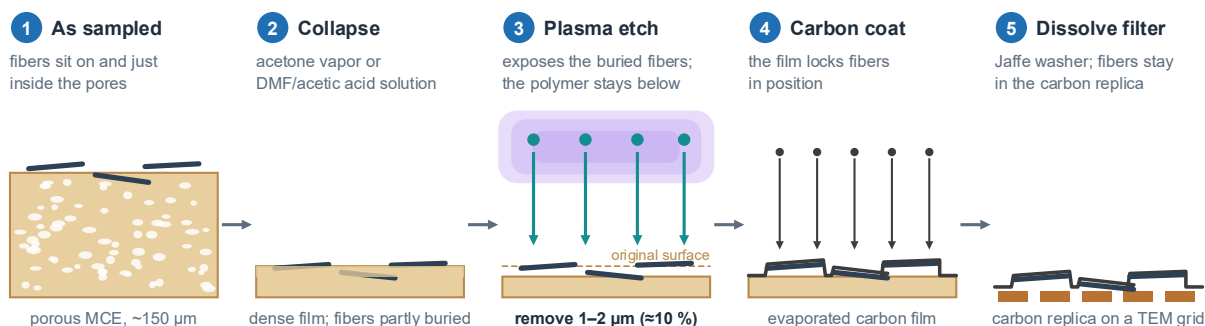
Remote (downstream) mode. Here the plasma is generated in a separate inductively coupled source attached to the chamber, and the gas enters the chamber through it. Ions and electrons recombine before they reach the sample, so the sample sees mainly neutral oxygen atoms, no glow and almost no UV [44]. This gives the gentlest treatment and the least heating, and it suits surface work where ion bombardment must be avoided. The oxygen-atom density, and therefore the rate, falls from the inlet toward the pump, so sample position matters. The downstream source is an option on Tergeo and Tergeo-Plus.

Pulsed mode. The RF power can be switched on for a set fraction of each pulse period, from 100 % down to below 1 %, so that the average power is the set power times the duty cycle [44]. A filter-etching recipe can then run at a few watts on average with a stable discharge, which makes a 1–2 μm etch easier to time and repeat.

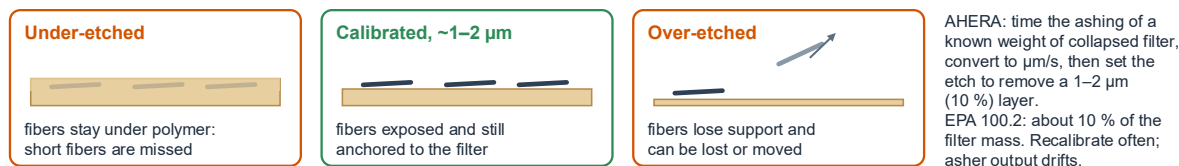
5 Applications

5.1 Asbestos in air and water: etching collapsed MCE filters

Direct-transfer TEM keeps every fiber where it landed on the filter, which preserves clusters, bundles and matrix particles in their original form. The price is a fussy preparation (Figure 6). The MCE filter is first collapsed with acetone vapor or a dimethylformamide solution, turning the porous membrane into a thin, dense film. Collapsing engulfs some fibers in polymer, so the surface is etched in an oxygen plasma, coated with evaporated carbon, and the remaining filter is dissolved away, leaving the fibers held in a carbon replica on a TEM grid [3,12].



Why the etch depth must be calibrated



Schematic, not to scale.

Figure 6. Direct-transfer TEM preparation of a mixed cellulose ester filter (AHERA, ISO 10312, ASTM D6281). The oxygen plasma removes only a 1–2 μm surface layer of the collapsed filter, exposing buried fibers while they are still held by the polymer below. Too little etching hides short fibers; too much frees them to move or be lost.

The AHERA method makes the etch mandatory and is unusually candid about why it cannot give a recipe: "Because plasma ashers vary greatly in their performance, both from unit to unit and between different positions in the asher chamber, it is difficult to specify the conditions that should be used. Insufficient etching will result in a failure to expose embedded filters [sic], and too much etching may result in loss of particulate from the surface." Instead, the laboratory times the ashing of a known weight of collapsed filter, converts the result to an etch rate in $\mu\text{m/s}$ and sets the time to remove "a 1–2 μm (10 percent) layer of collapsed surface" [3]. The asher should be supplied with oxygen and fitted with a throttle or bleed valve so that pumping and venting do not disturb the filter, and quality control includes a blank run beside a positive sample to check for carryover [3]. EPA's 1989 laboratory guidelines offer an equivalent performance test: set conditions so that an uncollapsed 0.45 μm MCE filter oxidizes completely in about 15 min; for one filter brand, about 8 min on the collapsed filter then gave satisfactory replicas [45]. NIST's accreditation handbook asks for a calibration curve of weight against ashing time for collapsed MCE filters and allows a shallower etch if a full 10 % roughens the replica [46], and EPA's current data-review guidance requires that calibration initially and quarterly, with a target of 5–10 % removal [47]. ISO 10312 and ASTM D6281 follow the same principle for ambient air [12,13], and EPA Method 100.2 for drinking water requires etching of 0.22 μm MCE filters, recommends it for 0.1 μm filters, and targets about 10 % of the filter mass [14].

Several studies have measured the effect of etch time. In an EPA bench study with a calibrated asher, each doubling of the etch time between 2 and 16 min added on average 13 % to the density of chrysotile structures longer than 0.5 μm , while counts of structures longer than 5 μm did not change [48]. Webber and co-workers found that the etch depth has to be calibrated at different positions in the chamber and that both over- and under-etching can lose short fibers [49]. Etch times of 6–14 min had no effect on measured densities in another comparison, except for fibers shorter than 1 μm deposited by liquid filtration [50]. In each study it was the short-fiber counts that moved with the etch.

Sample temperature and power control matter here. The collapsed film is thin (AHERA's 1–2 μm is a tenth of it) and the filter is rated to 75 $^{\circ}\text{C}$, so the plasma must remove polymer without softening or distorting it. A

low continuous power, or pulsed operation at a reduced duty cycle, stretches the etch to several minutes so that small timing errors matter less, and a low-recombination quartz chamber helps keep the oxygen-atom supply even across a batch, although each position should still be calibrated. Downstream mode removes ion bombardment entirely and is worth evaluating where replica texture is a problem.

5.2 Asbestos: full ashing for indirect-transfer TEM

When a filter is overloaded with dust or carries a lot of organic debris, direct transfer fails; ASTM D6281 notes that the direct method is not usable above roughly 10 % filter coverage [13]. ISO 13794 then prescribes an indirect route: "A portion of the filter is ashed in an oxygen plasma, and the residual ash is dispersed in distilled water with adjustment of the pH to between 3,0 and 4,0 using acetic acid," after which aliquots are filtered onto a polycarbonate filter of 0.2 μm or less or a cellulose-ester filter of 0.22 μm or less for TEM [15]. The first lane of Figure 7 shows the sequence. EPA's activity-based air sampling procedure uses the same approach, ashing the filter "until complete calcination" before recovering the ash in water [51].



Figure 7. Preparation routes that use full plasma ashing: indirect-transfer TEM for asbestos (ISO 13794), redeposition XRD for respirable crystalline silica (NIOSH 7500, with the FTIR variants of NIOSH 7602, 7603 and MSHA P-7), and mineral-matter analysis of coal.

Here the plasma's job is complete removal of the filter and the organic debris without touching the fibers. The redispersion step, not the ashing, is what changes the fiber population: ultrasonic dispersion breaks clusters and matrix particles into individual fibers and bundles, and some fibers fragment, so indirect counts are usually higher and shifted toward shorter fibers [15,52,53]. In a paired comparison, indirect results averaged 3.3 times the direct results, a difference that was not statistically significant across 31 samples [54]. Because the ash from a large area of sample filter ends up on a small analytical filter, any fiber contamination in the filter medium or the asher is concentrated too [51], so ash blanks and a clean chamber are part of the method.

Many laboratories ash indirect samples in a capped glass vial with a perforated foil lid, so that the residue can be redispersed in the same vessel. Our vial test (Section 6.5) shows the trade-off: the vial keeps everything

contained, but the narrow opening and the foil cut the oxygen-atom supply, and the ashing takes hours rather than minutes.

5.3 Asbestos in bulk materials and tissue

Resilient floor tile, mastic, asphalt roofing and similar non-friable organically bound (NOB) materials are the classic case where the organic matrix hides the fibers. The EPA bulk method notes that floor tiles "may contain fibers too small to be resolved by PLM ($< 0.25\ \mu\text{m}$ in diameter)" and that many mastics dissolve in refractive-index liquids [7]. Its gravimetric reduction removes the matrix by ashing, followed by acid dissolution, and tracks the mass at each step; asbestos content is then the product of the retained fractions and the asbestos fraction of the final residue, as in its worked floor-tile example of 70 % ash residue $\times$ 20 % acid residue $\times$ 50 % asbestos = 7 % [7]. The method allows a muffle furnace at 300–500 °C, with 6 h at 450 °C usually sufficient, and names the low-temperature plasma asher for small cellulosic samples and as an alternative for organic binders, cellulose and synthetic fibers, for which the method lists the furnace as the preferred route [7]. New York's ELAP methods ash NOB samples at $485 \pm 5\ ^\circ\text{C}$ before TEM and state that only TEM can show such a material to be non-asbestos [20]. The 2014 edition of ISO 22262-2 removes organic material at $450 \pm 10\ ^\circ\text{C}$ [55].

These furnace temperatures sit just below chrysotile's dehydroxylation and inside the range where crocidolite oxidizes (Figure 3a), which is why the methods cap them so carefully, and why New York's method for vermiculite-bearing materials requires refractive-index liquids for asbestos in both its natural and its "altered (heated or acid-stressed)" state [20]. Plasma ashing avoids that margin. It is most practical for small, thin or cellulose-rich samples: paper and felt backings, paint chips, sheet vinyl, and dust or wipe samples, where the organic mass is in the milligram-to-gram range that a tabletop asher handles in hours.

Lung tissue is the other bulk matrix where plasma ashing has a long record. Gylseth and co-workers freeze-dried tissue and ashed it below 200 °C at about 50 W with an oxygen flow of 225 sccm, completing it in 2–4 h; above about 150 W, local overheating caused significant fiber losses [31,32]. Manke and co-workers found no change in the length, diameter or aspect-ratio distribution of crocidolite after freeze-drying and plasma ashing, with at least 40 % of the fibers recovered [56]. A 2025 validation used 24 h of plasma ashing at 60–80 W after freeze-drying, checked against certified reference lung materials, and found no significant change in the morphology or X-ray spectra of the fibers [57]. Not every comparison favors ashing: in lung tissue from a patient with asbestosis, LTA yielded about twice as many fibers as wet digestion, and shorter ones, which has been attributed to long fibers breaking during preparation [58]. Low power, a cool sample and gentle handling of the residue reduce such losses.

5.4 Respirable crystalline silica

Silica methods use full ashing for a different reason: the PVC collection filter and any organic dust would interfere with X-ray diffraction or infrared measurement of quartz and cristobalite. NIOSH 7500 places the filters in 50-mL beakers in the low-temperature asher, adds 15 mL of 2-propanol after ashing, disperses the residue ultrasonically and redeposits it on a $0.45\ \mu\text{m}$ silver membrane for XRD; the alternatives are 2 h in a muffle furnace at 600 °C (800 °C if graphite is present) or dissolving the filter in tetrahydrofuran [8]. NIOSH 7602 ashes PVC filters in aluminum dishes and presses the residue into a KBr pellet for FTIR [21]. For coal mine dust, NIOSH 7603 specifies 2 h at 300 W and 75 sccm of oxygen before redeposition and FTIR [22], and MSHA's method P-7 ashes samples "in a low-temperature radio-frequency (RF) generated oxygen-plasma asher to remove the organic matrix and media", in about 60 min [23].

The low temperature matters most when the dust contains calcite. Coal mines use limestone rock dust, and in a muffle furnace calcite reacts with quartz and lowers the result; NIOSH 7602 and 7603 therefore call for an acid wash before furnace ashing when calcite is present, at more than about 20 % of the dust loading in 7602, a step plasma ashing does not need [21,22]. Kaolinite is the opposite case: it survives LTA and absorbs near the quartz band, so LTA samples need a kaolinite correction [22]. Comparisons of the two ashing routes are mixed. A NIOSH laboratory study found 7.0 ± 1.3 % quartz after plasma ashing and 7.5 ± 2.1 % after furnace ashing in the same coal dust, with no significant difference [59,60]. A study of muffle-furnace preparation reported quartz recoveries of 49–104 % after furnace ashing and 89–108 % after plasma ashing [61], and NIOSH authors responded that the two preparations are regarded as equivalent [60]. Solvent preparation has its own problem with engineered stone: THF gave crystalline-silica results 21.5–30.9 % lower than furnace ashing for engineered-stone dust, which contains resin [62].

Ashing PVC releases chlorine-containing products such as HCl along with CO₂ and water, so the chamber and pump must tolerate them. A quartz chamber resists them, and Tergeo systems are offered with oxygen-rated dry pumps or PFPE-oil pumps [44]; the pump supplier's guidance on chlorinated by-products should be followed.

5.5 Coal mineral matter and coal-mine dust

Coal is where low-temperature ashing has been studied most thoroughly. Conventional ash tests burn coal at 750–815 °C, which decomposes carbonates, dehydrates clays and oxidizes sulfides, so the high-temperature ash is neither the same mass nor the same minerals as the mineral matter in the coal [9]. Ward describes LTA as probably "the most reliable method for determining the percentage of total mineral matter in higher-rank coals", and it is the basis of the Australian standard AS 1038.22 [9,24]. In the USGS interlaboratory study of the Herrin No. 6 coal, the five laboratories that reported LTA yields averaged 16.8 %, against a high-temperature ash of 13.4–14.0 % for the same coal; the difference is largely the water, carbon dioxide and sulfur that the furnace drives off [25]. Quartz, kaolinite and illite come through LTA unaltered, which is what makes the residue useful for XRD and Rietveld quantification [25].

Published conditions vary widely. The nine laboratories that reported their conditions in the USGS study ran 15–133 W per chamber and 60–800 sccm of oxygen, loaded 0.4–4 g of coal per dish and ashed for 0.5 to 8 days with 4–12 stirrings [25]. Miller recommended moderate power, a layer density below about 30 mg/cm² and stirring every 1–2 h, which gave a quantitative ash in 12–15 h, and defined the end point as a mass change of less than 2 mg per 2-hour period [25]. Han and co-workers settled on 200 W, 150 sccm and 500 mTorr, at a chamber temperature of 121 °C, as the best balance between complete carbon removal and preserved clay structures [30]. Pike and co-workers showed that a residue ashed to constant mass can still hold organic material, so grinding and re-ashing may be needed for quantitative work [63].

Coal-mine dust is the occupational side of the same problem: the quartz in respirable coal-mine dust is measured by FTIR after low-temperature ashing of the PVC filter (NIOSH 7603, MSHA P-7) [22,23].

5.6 Other environmental uses

Plasma ashing also appears in several smaller applications:

- **Man-made mineral fibers.** Refractory ceramic fibers are recovered from lung tissue by low-temperature ashing in retention and biopersistence studies [64].
- **Inorganic fibers by SEM.** ISO 14966 treats gold-coated capillary-pore filters in a plasma asher to remove organic particles before SEM-EDS counting [16].

- **Biogenic silica.** Low-temperature plasma ashing was the most suitable way to remove organic matter from diatom frustules in short-term dissolution studies, with the least effect on dissolution [65].
- **Sediments.** Plasma oxidation removed the most organic carbon from an estuarine sediment standard and from one of two field sites, and conditions can be chosen to limit pyrite oxidation [66].
- **Trace elements.** LTA retains most elements at better than 95 %, but recoveries of arsenic, cadmium and selenium were 55–94 % and those of mercury, bromine and iodine were also lower in one systematic study [42]; recovery should be validated for each analyte.

Plasma ashing is the wrong tool when the organic material is the analyte. It destroys microplastics, pollen walls and organic contaminants along with the matrix.

6 PIE Scientific test results

6.1 Equipment, materials and measurements

The tests used Tergeo-series tabletop plasma systems (a Tergeo-Plus for the covered-dish and vial runs) running at 13.56 MHz in direct mode with continuous RF power (pulse ratio 255/255). The Tergeo has a quartz chamber of 110 mm inner diameter and 280 mm depth; the Tergeo-Plus chamber is 160 mm by 280 mm [44]. Gas flows were set by the built-in mass-flow controllers, chamber pressure was read by the Pirani gauge, and the plasma emission intensity was logged by the built-in sensor once per second. Slow venting was used to keep the light ash in place.

The filter tests used Tisch SF15103 cellulose acetate membranes, 47 mm in diameter with 0.45 μm pores [67], in glass Petri dishes. Cellulose acetate is one of the two esters in MCE filters, but it is a different product from the MCE filters named in AHERA, so these results show filter removal rates and temperatures rather than an AHERA etch calibration. The vial test used a 25 mm membrane filter in a glass vial capped with perforated aluminum foil, following a commercial asbestos laboratory's indirect-preparation procedure. The bulk tests used about 1 g each of charcoal powder and three coal powders (T-4, Z-4 and Z-5) spread in glass Petri dishes, and Kimberly-Clark laboratory wipes ashed in argon plus oxygen.

Temperatures were measured with a thermocouple in contact with the underside of the glass dish. Coal masses were weighed with the dish and corrected for its tare. Times are given from plasma ignition. We read the emission end point from the logs after each run, as the time at which the 31-s rolling median of the emission signal had fallen 90 % of the way from its plateau to its final baseline; during a run, the operator sees the same fall on the screen.

6.2 Cellulose acetate membrane filters

Both filters disappeared completely, leaving only a few white flakes of inorganic residue so light that a gentle air current would carry them away (Figure 8). At 30 W and 90 sccm O_2 (about 1,000 mTorr), ashing took about 20 min by eye and the emission end point came at 15.5 min. At 15 W the same filter needed about 60 min, with the emission end point at 55.5 min (Table 2, Figure 9). Halving the power roughly tripled the ashing time, which gives a laboratory a simple way to slow the process when it wants more control.

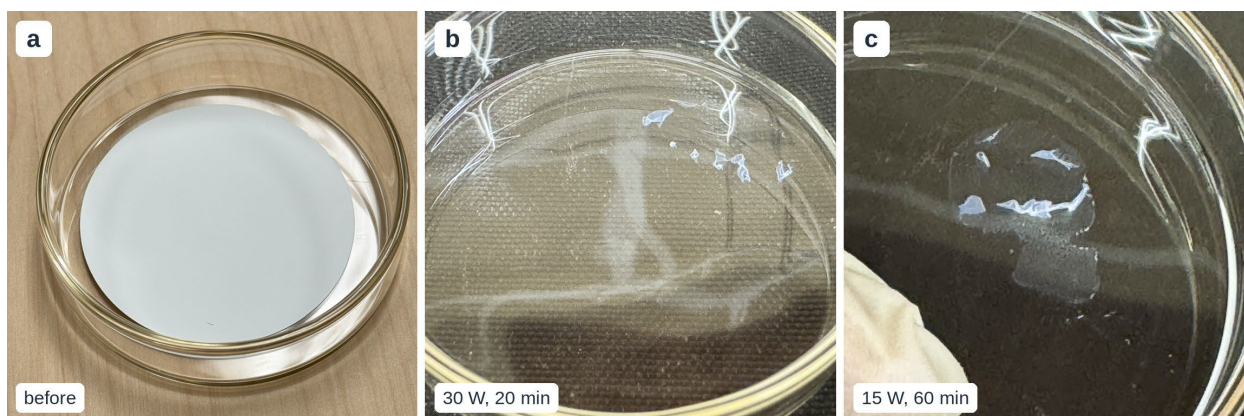


Figure 8. Tisch SF15103 cellulose acetate membrane filters (47 mm, 0.45 μm) in glass Petri dishes. (a) Before ashing. (b) After 20 min at 30 W and 90 sccm O_2 . (c) After 60 min at 15 W and 90 sccm O_2 . Only a few white flakes of inorganic residue remain.

Table 2. Membrane filter ashing in Tergeo-series systems (direct mode, continuous power, oxygen).

Sample and holder	RF power	O_2 flow	Pressure	Ashing time (visual)	Emission end point	ΔT under dish
47 mm cellulose acetate filter, open glass dish	30 W	90 sccm	$\approx 1,000$ mTorr	≈ 20 min	15.5 min	+8 $^\circ\text{C}$
47 mm cellulose acetate filter, open glass dish	15 W	90 sccm	not recorded	≈ 60 min	55.5 min	+6 $^\circ\text{C}$
47 mm filter, dish under perforated Al foil (Tergeo-Plus)	75 W	90 sccm	830 mTorr	≈ 20 min	not logged	not measured
25 mm filter in foil-capped glass vial (Tergeo-Plus)	75 W	90 sccm	830 mTorr	1–2 h	not logged	not measured

Times are from plasma ignition. Emission end point: time at which the smoothed emission signal had fallen 90 % of the way from plateau to baseline. ΔT : rise recorded by a thermocouple under the glass dish during the run. The vial result is the range from repeated runs.

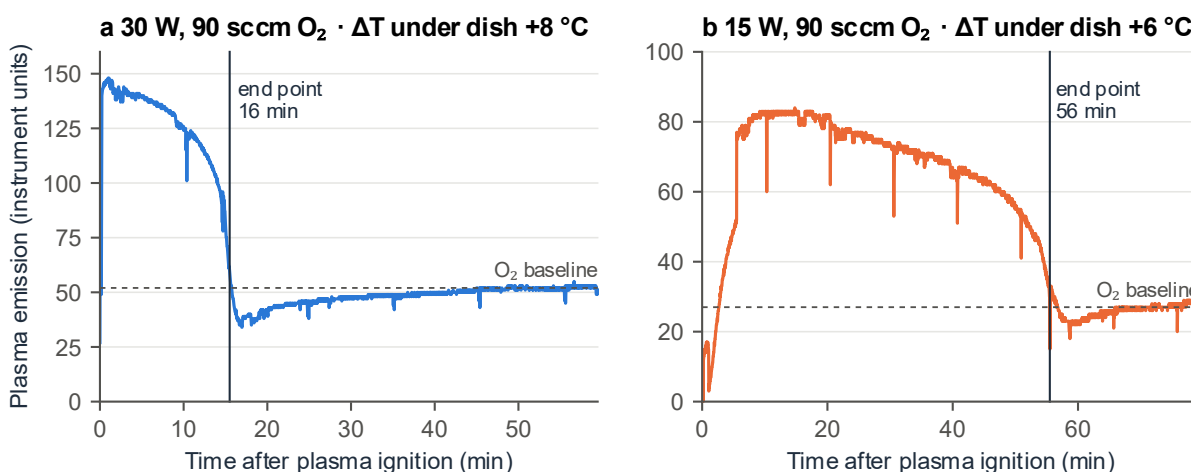


Figure 9. Plasma emission during ashing of the 47 mm cellulose acetate filters. The signal is high while CO , CO_2 and H_2O are being produced and falls to the pure-oxygen baseline when the filter is gone. Times are from plasma ignition; vertical lines mark the emission end points at 15.5 min (30 W) and 55.5 min (15 W). Both runs continued after the end point, and the logs are shown in full.

6.3 Sample temperature

The thermocouple under the dish recorded a rise of 8 °C in the 30 W filter run and 6 °C in the 15 W run; the plasma stayed on for about 60 and 80 min in those runs, and the time of the reading was not logged (Figure 10). In a run at 75 W and 10 sccm O₂ with carbon powder in the dish, the same thermocouple read 27.4 °C at ignition and 43.1 °C after 15 min, a rise of 16 °C that had not yet leveled off when the record ended.

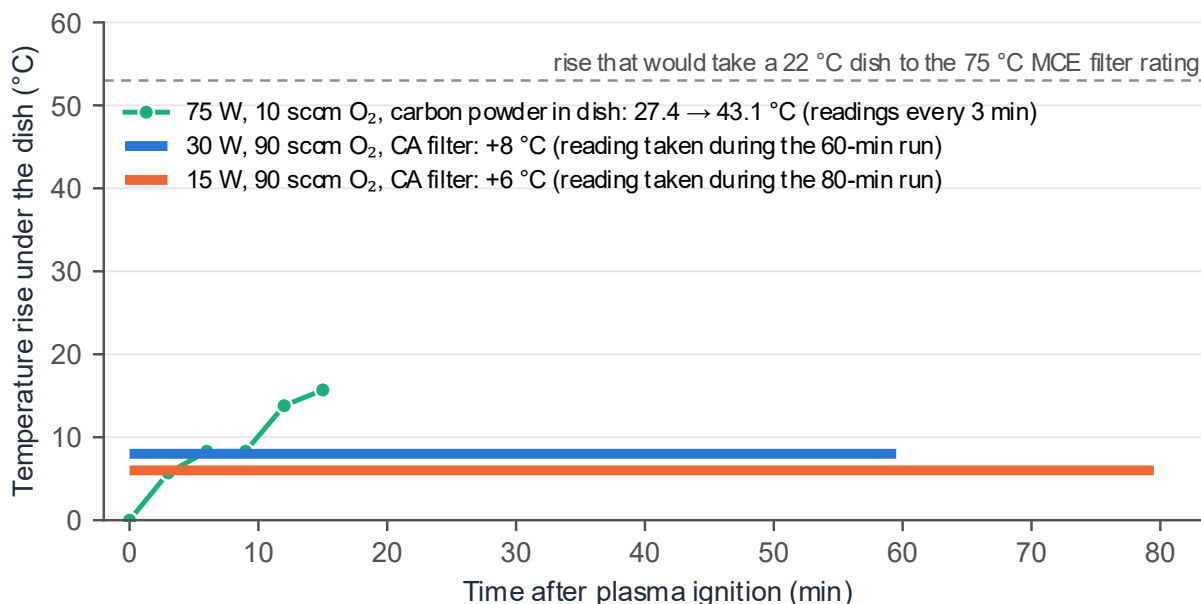


Figure 10. Temperature rise measured by a thermocouple under the glass dish in Tergeo systems. For the 15 W and 30 W filter runs, the recorded rise is drawn across the full plasma-on time because the time of the reading was not logged; the 75 W run with carbon powder is shown for its first 15 min, read every 3 min. The dashed line marks the rise that would take a dish starting at 22 °C to the 75 °C rating of an MCE filter.

A reading under the glass is a lower bound for the membrane surface, so it does not by itself prove that a filter stays below its 75 °C rating. It does show how little heat these recipes put into the dish: a few degrees at 15–30 W, compared with 88–177 °C chamber temperatures reported for a larger asher at 100–400 W [30] and the 100–200 °C typical of coal LTA [25]. Han and co-workers also measured 123 °C at 75 W in another tabletop unit, so power alone does not set the temperature; chamber size, gas flow, sample load and holder all play a part. We have not yet logged the temperature through a full multi-hour 75 W coal run. A laboratory that needs a documented sample temperature can place an irreversible temperature label or a fiber-optic probe at the sample position (Section 7).

6.4 End-point detection from the plasma emission

The Tergeo plasma sensor reads the visible emission from the glow. The sensor does not separate wavelengths, but pure oxygen emits weakly while the oxidation products make the glow much brighter, so the signal rises as soon as ashing starts and falls back to the oxygen baseline when the organic material is gone (Figures 9 and 12). The ratio of plateau to baseline was about 3:1 for the filters and 7:1 to 12:1 for coal and charcoal, large enough to read without any calibration. Because the signal is logged with the recipe, the end point is documented for every sample and the chamber need not be opened to check progress.

The coal runs also show what the signal cannot tell. When Z-5 was stirred after 4 h, the signal jumped from about 2,800 to 3,600 units as fresh coal was brought to the surface, and a second stirring near 6 h produced

no rise, which confirmed the end point. In Z-4 the signal had been at its baseline for 11 h; stirring produced only a small, short rise, yet a further 5.7 h of ashing removed another 11 mg, 1.3 % of the original mass. Organic material sealed under a crust of ash contributes little to the emission. For gravimetric work, the emission end point tells the analyst when to stir and weigh, and constant mass after stirring confirms completion, as Miller recommended [25].

6.5 Covered dishes and vials

In the Tergeo-Plus at 75 W and 90 sccm O₂ (830 mTorr), a 47 mm filter in a Petri dish covered with perforated aluminum foil was ashed in about 20 min (Figure 11a–c). With slow pumping and slow venting enabled, the foil can be left off, with the power reduced to about 30 W to slow the reaction. A 25 mm filter at the bottom of a foil-capped glass vial took much longer: in our runs the filter was gone within 1–2 h, and photographs of one run show it present at loading and gone 2 h 9 min later (Figure 11d–f). The laboratory that supplied the procedure reported that its current process needed an overnight run.

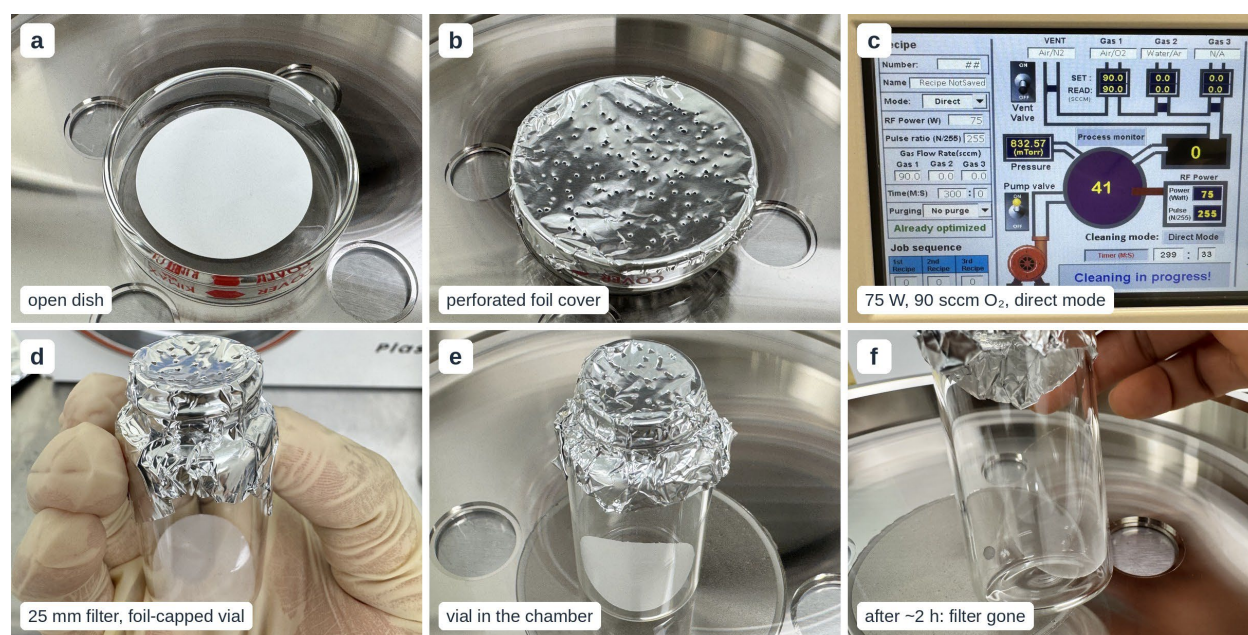


Figure 11. Sample configurations tested in the Tergeo-Plus. (a) Filter in an open Petri dish. (b) The same dish under perforated aluminum foil. (c) Recipe screen: 75 W, 90 sccm O₂, 832 mTorr, direct mode, continuous power. (d) A 25 mm filter in a glass vial capped with perforated foil. (e) The vial in the chamber. (f) The same vial 2 h 9 min after (e): the filter has been ashed.

The slower rate in the vial is what Section 3.3 predicts. Oxygen atoms have to pass through a few pin holes in a metal foil and diffuse to the bottom of a narrow tube, losing a share to the foil and the glass walls on the way. Wider and shallower vessels, larger or more numerous holes, or a glass cover instead of foil should shorten the run, as should ashing the filter in an open dish with slow venting and transferring the residue afterwards, where the method allows it. We have not yet tested these variants.

6.6 Coal and charcoal

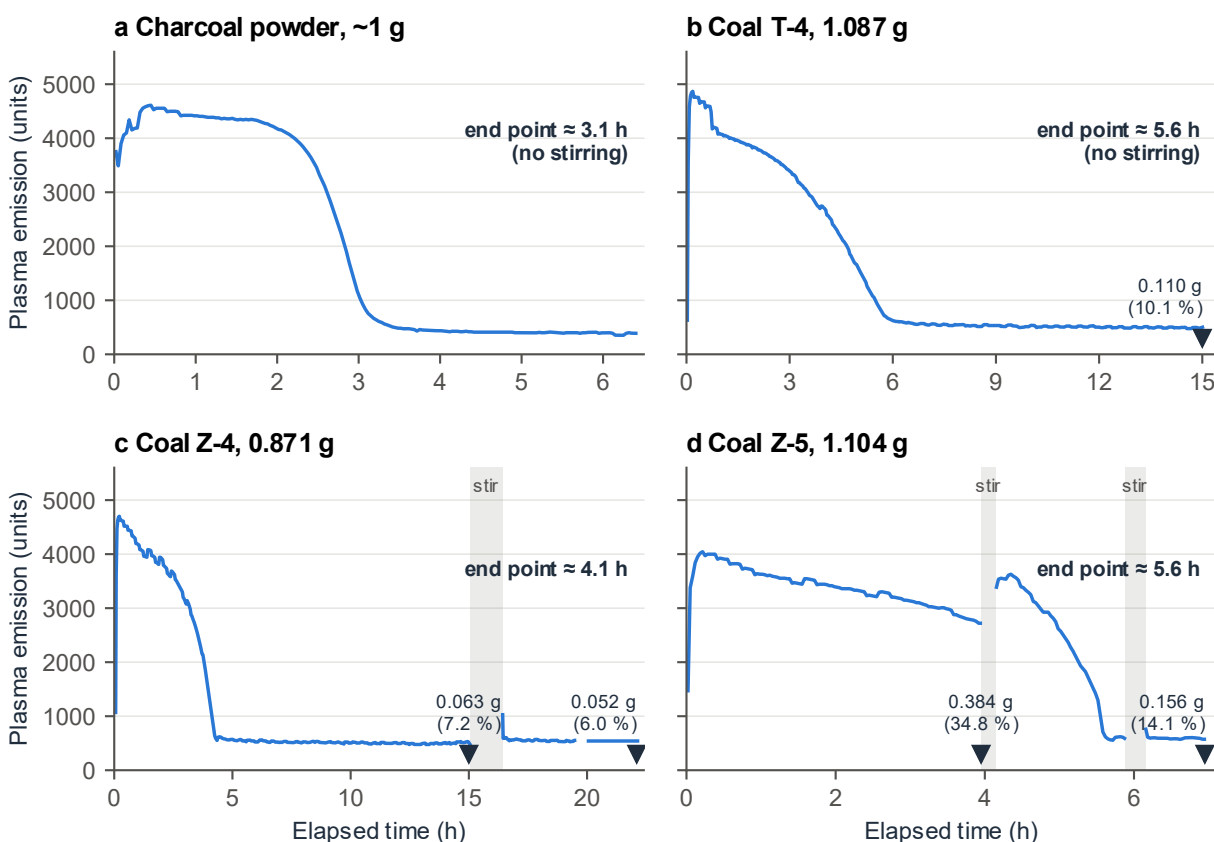
Coal and charcoal are a harder test than a filter: about a gram of carbon spread over the bottom of a Petri dish. At 75 W and 10 sccm O₂ (140–240 mTorr), all four samples ashed to a mostly light gray or white residue, with a few dark streaks of unreacted coal left in Z-4 (Table 3, Figures 12 and 13). Charcoal and T-4 were left untouched for the whole run; Z-4 and Z-5 were stirred and weighed part way. Charcoal reached its emission

end point after 3.1 h. The three coals reached theirs after 4.1–5.6 h and left 6.0–14.1 % residue. These are LTA residues, not standard ash values, and no reference ash was measured for these coals. T-4 was never stirred and still ashed from 1.087 g to 0.110 g (10.1 %) in a 15 h unattended run.

Table 3. Ashing of charcoal and coal powders (Tergeo series, direct mode, 75 W, 10 sccm O₂, 140–240 mTorr).

Sample	Initial mass	Treatment	Emission end point	Residue	Residue, % of initial
Charcoal powder	≈1 g	6.4 h, no stirring	3.1 h	not weighed	–
Coal T-4	1.087 g	15 h, no stirring	5.6 h	0.110 g	10.1 %
Coal Z-4	0.871 g	15 h, no stirring	4.1 h	0.063 g	7.2 %
Coal Z-4		then stirred, ≈5.7 h more	small, short rise only	0.052 g	6.0 %
Coal Z-5	1.104 g	4 h, no stirring	–	0.384 g	34.8 %
Coal Z-5		then stirred twice, ≈3 h more	5.6 h	0.156 g	14.1 %

Masses are corrected for the dish tare. The Z-5 record notes possible loss of ash during stirring and some large particles that were not fully ashed. Emission end points are elapsed times, defined as in Table 2.



75 W, 10 sccm O₂, 140–240 mTorr, direct mode. ▼ = weighing (residue mass, % of initial). Shaded = plasma off to stir and weigh.

Figure 12. Plasma emission during ashing of charcoal and three coal powders at 75 W and 10 sccm O₂. Each trace falls to the oxygen baseline when the carbon in the layer has been oxidized. Triangles mark weighings, with the residue mass and its percentage of the initial mass. Gray bands mark interruptions for stirring and weighing; in Z-5, the first stirring exposed fresh coal and the signal rose again.

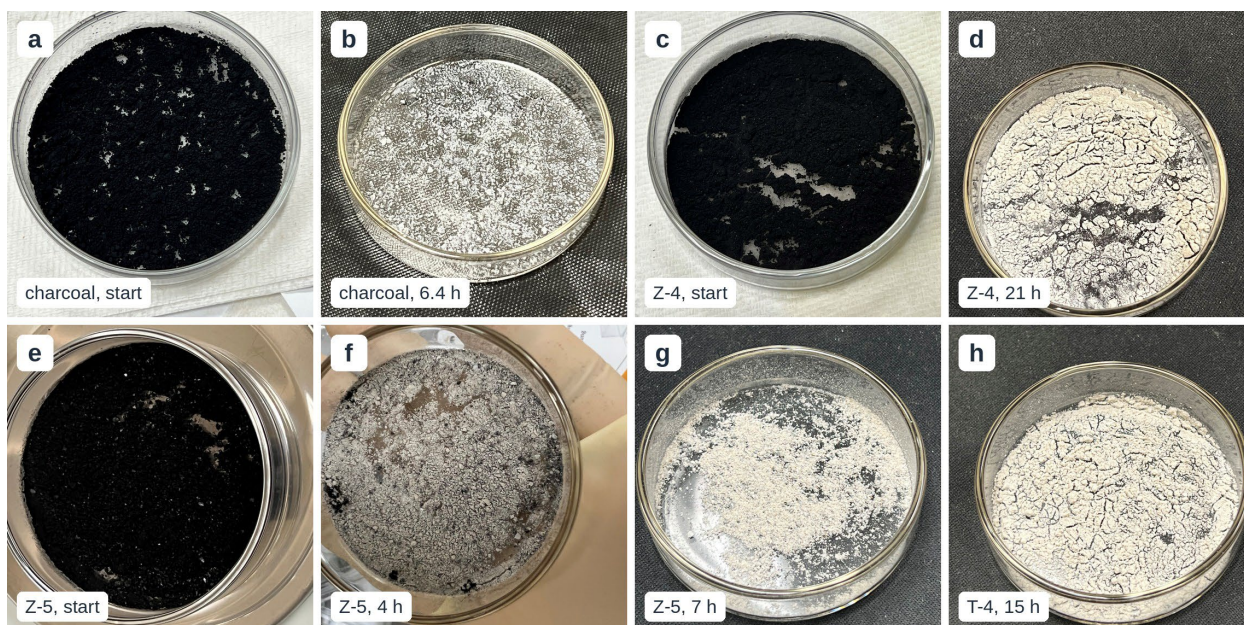


Figure 13. Charcoal and coal powders before and after plasma ashing at 75 W and 10 sccm O₂. (a, b) Charcoal before and after 6.4 h. (c, d) Coal Z-4 before and after about 21 h of ashing. (e–g) Coal Z-5 before, after 4 h, and after 7 h with stirring. (h) Coal T-4 after 15 h without stirring. The dark streaks in (d) are where ash had crusted over unreacted coal.

For comparison, the USGS laboratories took 0.5–8 days, with up to 12 stirrings, to bring comparable loads to constant mass [25]. Our runs reached the emission end point in 3–6 h, which is not the same criterion, but it shows how quickly most of the carbon goes. The Z-4 and Z-5 histories show why stirring still belongs in a quantitative procedure: in Z-5 the first stirring exposed coal that had been shielded by ash, and in Z-4 it released 11 mg that the emission signal no longer registered. A thinner layer, a stir at the first emission end point and a final weighing to constant mass would give a defensible LTA yield.

6.7 Cellulose wipes

Laboratory wipes stand in for cellulose-rich samples such as wipe samples, paper and felt. Ashed overnight in argon plus oxygen (power and flows were not recorded for this run), the wipes left a small, porous mat of inorganic residue (Figure 14). The color of the glow tracked the reaction: it was whitish while water and CO₂ were being released and turned violet, the color of the argon-oxygen plasma itself, when the ashing was complete. As with the filters, slow venting kept the residue in the dish.

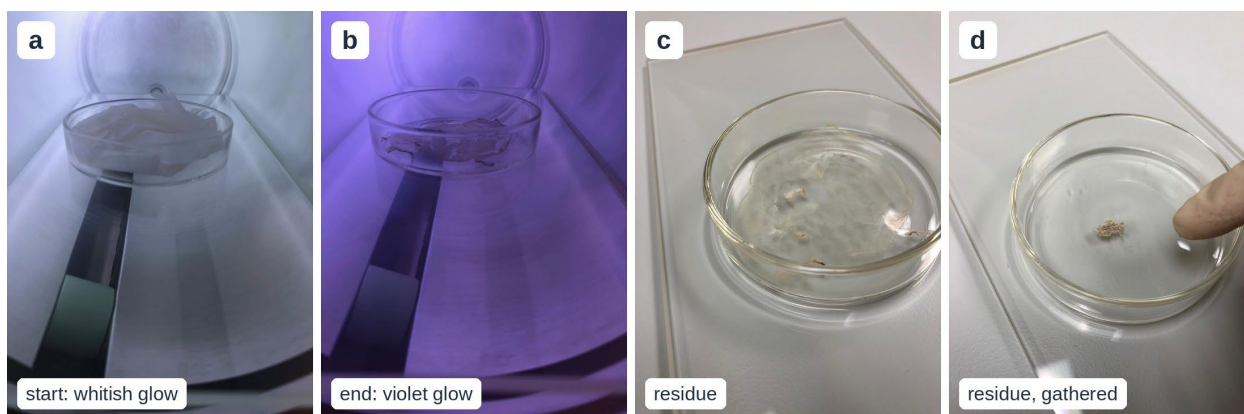


Figure 14. Kimberly-Clark laboratory wipes ashed overnight in argon plus oxygen. (a) Start of the run: the glow is whitish from the water and CO₂ being released. (b) End of the run: the violet glow of the argon-oxygen plasma shows that oxidation is complete. (c, d) The inorganic residue left in the dish.

7 Practical guidance

Table 4 collects starting conditions from this work and from the methods cited above. They are starting points for a laboratory's own calibration, not substitutes for it; AHERA, NIST and EPA all require each asher to be calibrated in place [3,46,47].

Table 4. Starting conditions for Tergeo systems, with the checks each application needs.

Application	Mode	Starting conditions	Checks
Etching collapsed MCE filters (AHERA, ISO 10312, EPA 100.2)	Direct at low power, or pulsed; downstream worth evaluating	Pure O ₂ ; choose power and time so that 1–2 μm ($\approx 10\%$ by weight) is removed over several minutes; slow pumping and venting	Weight-loss calibration with collapsed filters at sample positions, repeated quarterly; replica quality; carryover blank [3,47]
Full ashing of filters (ISO 13794; NIOSH 7500, 7602, 7603)	Direct	47 mm cellulose acetate filter: 30 W, 90 sccm O ₂ , ≈ 20 min, or 15 W, ≈ 60 min (PIE). PVC filters not yet tested; expect different times	Ash blank for each filter lot; complete transfer of residue; slow venting
Filters in vials for indirect preparation	Direct, with perforated cap	25 mm filter: 75 W, 90 sccm O ₂ , 1–2 h (PIE); wider vessels or more holes shorten the run	Visual check for residue; blank vial
Coal, charcoal and organic bulk	Direct	≈ 1 g per dish: 75 W, 10 sccm O ₂ , emission end point in 3–6 h (PIE); layer below ≈ 30 mg/cm ²	Stir at the first end point; ash to constant mass, < 2 mg change per 2 h [25]
Lung and other tissue	Direct, low power	Freeze-dry first; keep power moderate with ample O ₂ flow (published work used ≈ 50 –80 W) [32,57]	Reference materials; fiber size distributions
Trace elements in the residue	Direct	As for the matrix	Spike recoveries for each element, especially As, Cd, Se and Hg [42]

Conditions from this work are marked (PIE). All conditions are starting points; each laboratory must calibrate its own asher and validate recovery for its analyte.

Some practices apply to every application:

- **Calibrate where the samples sit.** Ash rate varies with position, so calibrate with filters at the positions used for samples and repeat the calibration on a schedule, quarterly for TEM laboratories [47,49].

- **Control pumping and venting.** Ash from filters and wipes is light enough to be blown off a dish. Use slow pumping and slow venting; New York's ELAP audit checklist asks for a vent time of at least one minute [68].
- **Use pure oxygen for TEM work and run blanks.** Run a blank filter beside positive samples to check for carryover, and run ash blanks for each new lot of filters [3,8].
- **Document the sample temperature once.** Place an irreversible temperature label, or a thermocouple or fiber-optic probe, at the sample position under the laboratory's own recipe and record the reading with the method validation.
- **Stir and weigh bulk samples.** Spread coal and bulk samples below about 30 mg/cm², stir at the first emission end point and ash to constant mass [25].
- **Treat the residue as hazardous.** Ashing removes the organic matrix but leaves the asbestos or silica, now in a light, easily dispersed form. Load and unload in a suitable hood and filter the pump exhaust.

The choice of model depends mainly on batch size. The Tergeo, with a 110 mm chamber, suits a few filters per run. The Tergeo-Plus (160 mm) and Tergeo-Pro (230 mm) hold larger batches of dishes, slides or beakers, with RF power options up to 500 W for heavy bulk loads [44]. Direct-mode and pulsed operation are available across the series, and the downstream source is an option on the Tergeo and Tergeo-Plus.

8 Conclusions

Low-temperature oxygen plasma ashing removes organic matter by chemistry rather than heat. Oxygen atoms made by electron collisions oxidize filters, binders, tissue and coal to CO, CO₂ and H₂O, and the sample stays far below the temperatures at which asbestos, quartz, clays and carbonates change. That is why the technique appears in the AHERA, ISO, EPA, NIOSH and MSHA methods for asbestos and crystalline silica and in coal mineralogy. One instrument does three jobs: a 1–2 µm etch of a collapsed MCE filter, complete ashing of a filter, and removal of grams of organic matrix. Each needs its own calibration.

In Tergeo systems, a 47 mm cellulose acetate filter was ashed in about 20 min at 30 W or 60 min at 15 W, with a temperature rise of 6–8 °C under the dish. A filter in a foil-capped vial was ashed in 1–2 h at 75 W, and gram-scale coal and charcoal samples reached their emission end points in 3–6 h, three of the four without any stirring. The plasma emission signal showed the end point in every logged run, and stirring with a final weighing confirmed it for coal. The next step for a laboratory is a recovery study with its own filters, reference materials and sample positions; PIE Scientific can run such tests on request.

Frequently asked questions

What is low-temperature plasma ashing? It is the removal of organic material by oxygen atoms made in a low-pressure RF discharge. The organic matter leaves as CO, CO₂ and H₂O, and the inorganic part of the sample stays behind, while the sample remains far cooler than in a muffle furnace.

How hot does the sample get? Published coal-ashing work reports 100–200 °C, and chamber temperatures of 88–177 °C at 100–400 W have been measured in a commercial asher [25,30]. In our Tergeo tests the dish underside rose by 6–8 °C during membrane-filter runs at 15–30 W.

Why do AHERA and ISO 10312 require plasma etching of MCE filters? Collapsing the filter buries some fibers in polymer. A plasma etch of 1–2 μm exposes them without freeing them from the filter, so short fibers are counted [3,12].

Is NIOSH 7402 prepared with plasma ashing? No. Ashing destroyed the organic fibers that the method counts in its denominator, so the step was removed [18,19].

Can plasma ashing replace a muffle furnace for silica analysis? NIOSH 7500, 7602 and 7603 accept either, and NIOSH authors regard the two as equivalent. Plasma ashing avoids the calcite–quartz reaction that a furnace can cause in coal-mine dust [8,22,60].

How long does ashing take in a Tergeo? In our tests a 47 mm cellulose acetate filter took about 20 min at 30 W, a 25 mm filter in a capped vial 1–2 h at 75 W, and about 1 g of coal 4–6 h at 75 W to the emission end point.

About PIE Scientific

PIE Scientific LLC designs and builds plasma, ion and electron beam equipment in Union City, California. The Tergeo, Tergeo-Plus and Tergeo-Pro tabletop plasma systems are used for plasma ashing, etching, cleaning and surface treatment. To discuss an environmental or occupational ashing application, or to send samples for a test, contact PIE Scientific through www.piescientific.com.

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