

## Cleaning large optics with CO<sub>2</sub> snow

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### ABSTRACT

The theory, methods, requirements, limitations, effectiveness and economics of cleaning large telescope mirrors with carbon dioxide (CO<sub>2</sub>) snowflakes are discussed. The method holds promise as a rapid, easy-to-use technique for routinely maintaining the cleanliness of mirrors between washings.

### 2. DEFINITION OF THE PROBLEM

The cleaning of light scattering particles and debris from large telescope mirrors is a challenging problem. Dirt may range in size from microscopic particles (Fig 1) to debris which includes leaves, insects, and man-made objects (Fig 2). Some foreign material simply sits on top of the reflecting surface and can easily be removed by tipping the mirror or lightly blowing across its surface. Other materials may adhere to the mirror by either physisorption or chemisorption. Physisorbed submicron-sized particles may attach themselves to an aluminum mirror surface with a force of more than 100g's<sup>1</sup>. Such forces make small particles difficult to remove. Chemisorbed mirror contamination includes particles attached to the surface of the mirror by drops of dome condensation moisture and its solutes falling onto the mirror, as well as materials "glued" to the mirror by the bodily fluids of insects. Fig 3 shows spots left behind by condensation drops on one of the six mirrors of the Multiple Mirror Telescope (MMT) on Mt Hopkins, Arizona. Sometimes chemical action involving water droplets and their solutes, the aluminum mirror surface, and previously deposited physisorbed particles results in severe localized mirror corrosion, some of which can be seen in Fig 4. Once a significant amount of chemisorbed dirt resides on the mirror, there are few options other than a solution washing of the mirror surface. However, between washings it would be valuable to have an easy-to-implement, rapid technique for removing loosely deposited surface debris and physisorbed particles. Not only would such a procedure reduce the optical background of scattered light, but it would also minimize mirror damage from chemisorption and corrosion by reducing the total mass of contaminant chemicals on the mirror surface which could attack the coating.

### 3. THEORY

The most obvious method of removing loosely held dirt is simply to blow across the surface. Consider an arbitrary stationary particle of mass  $M$  which is so small that it feels the effect of individual gas molecule impacts, each molecule having a mass  $m$  and a velocity  $v$ . This is exactly the situation experienced by micron-sized particles of contamination, since such particles are known to exhibit Brownian motion due to individual molecular impacts at STP<sup>2</sup>. Conservation of energy and momentum implies that in an elastic, head-on, collision with a single gas molecule a dirt particle will inherit a velocity  $V$  given by

$$V = [ 2 (m/M) v ] / [ 1 + m/M ] \quad (1)$$

Since for air molecules  $m/M \ll 1$ , the velocity  $V$  will be very small. Of course, it is only required that  $V$  be sufficiently large to move the dirt particle against fluid (air) drag to the next location to be cleaned. However, for small spherical particles the drag force may be computed from Stoke's law,<sup>3</sup>

$$F = 6 \pi \mu a V \quad (2)$$

where  $F$  is the drag force,  $\mu$  is the fluid (air) viscosity, " $a$ " is the particle radius, and  $V$  is the velocity of the particle contaminant through the air. A simple substitution of values for a micron-sized particle shows that this value can be quite large compared to  $Mg$ , where  $g$  is the acceleration of gravity. Therefore,  $V$  cannot be too small or else the particle's speed will be reduced to zero before it moves a significant distance. If the mass  $M$  is physisorbed, then the mass  $M$  in equation 1 must be replaced by an effective mass  $M_{\text{eff}}$  which is larger than  $M$ . The efficiency of the blowing method can, of course, be improved by increasing  $v$  and/or by using a heavy gas like xenon to increase  $m$ . Both of these tactics will reduce the number of particles left behind on the cleaned surface. Cleaning methods which

rely on momentum exchange with gasses are, however, limited by the extremely small mass of gas molecules

If snowflakes of carbon dioxide are used instead of gas molecules, then  $m$  can be made comparable to  $M$  with a concomitant increase in cleaning efficiency in spite of high contaminant particle adhesion and drag forces. Furthermore, CO<sub>2</sub> snowflakes can be made from very pure gas. Such flakes leave no residue behind on a surface to be cleaned since CO<sub>2</sub> sublimates directly to the gas phase. Carbon dioxide snowflake cleaning is also very gentle to the surface being cleaned because the flakes slide over the surface on a cushion of CO<sub>2</sub> gas which sublimates off flakes at an explosive rate. The effect is similar to that demonstrated by the classical experiment of the "dry ice puck".<sup>4</sup> Consequently, scratching of aluminum and gold coatings (Mohs 2.5) by CO<sub>2</sub> ice crystals (also Mohs 2.5) has never been observed. Explosive sublimation can be more quantitatively understood in terms of the Arrhenius relation.<sup>5</sup> The number of CO<sub>2</sub> molecules  $N$  capable of sublimation at Kelvin temperature  $T$  out of a total of  $N_0$  molecules is given by

$$N = N_0 e^{-E_s/kT} \quad (3)$$

where  $E_s$  is the energy of sublimation per molecule and  $k$  is Boltzmann's constant. Therefore, the number of subliming molecules grows exponentially above the sublimation temperature. Of course, even at room temperature the interior of a flake remains cold, since the thermal conductivity of an insulator like dry ice is very small. Nevertheless, at room temperature, CO<sub>2</sub> molecules will blow off the surface of the flake in shells of differential thickness thereby allowing the flake to skate across a surface layer of CO<sub>2</sub> gas. Complete sublimation of typical flakes has been measured to take place in less than 1 second at STP. When a flake of CO<sub>2</sub> snow collides with a particle, the jet-like CO<sub>2</sub> gas flux may wedge its way under a dislodged contaminant and carry it away from the surface. This view is supported by the complete lack of evidence of any kind of scratching by abrasive particles as they are removed from a surface (see the next section). Finally, it is important to realize that CO<sub>2</sub> snow cleaning imparts a very small thermal shock to a surface. This phenomena is easy to understand in terms of the heat conduction equation.<sup>6</sup>

$$H = K A (t_2 - t_1) / L \quad (4)$$

where  $H$  is the heat flow,  $K$  is the thermal conductivity,  $A$  is approximately the cross sectional area of the snowflake,  $t_2 - t_1$  is the difference in temperature between the flake and the surface, and  $L$  is the distance between the flake and the surface. Although  $L$  may be as small as 0.1 microns, as it must occasionally be to remove submicron-sized particles, such close encounters with the surface are to be expected only initially. A more likely value of  $L$  is the thickness of the gaseous boundary layer which may have a typical value of  $10^{-3}$  cm.<sup>7,8</sup> so that the value of  $A/L$  is approximately  $10^{-6} / 10^{-3}$  cm (or 0.001 cm). Since the temperature difference  $t_2 - t_1$  is about 100° C and the thermal conductivity of CO<sub>2</sub> gas is  $28 \times 10^{-6}$  cal/(sec cm<sup>2</sup>(°C/cm)),<sup>9</sup> it is clear that  $H = 2.8 \times 10^{-6}$  cal/sec. This is a very small rate of heat transfer per flake, however, many flakes reach the surface. Of the total amount of CO<sub>2</sub> used to produce flakes about half will end up in the solid state, the rest of the CO<sub>2</sub> goes into the gas phase.<sup>10</sup> Of the flakes that are formed, many miss the surface due to an incorrect initial velocity vector or turbulence, while others quickly sublime in transit. Only about 0.1% of the original CO<sub>2</sub> ends up as flakes which participate in cleaning and therefore enter into close proximity with the surface. This can be verified by allowing flakes to pile up on a grooved surface (such as a file) and measuring the weight difference of both the file and the CO<sub>2</sub> tank. For the gas flows used in experiments described in latter sections, a total of  $10^6$  flakes per second could be expected to bombard the surface. Therefore, thermal shock should be on the order of a degree Celsius per second. In fact, this is what has been observed, lending further support to the "dry ice puck" model of cleaning. It should also be noted that cooling from the cold CO<sub>2</sub> gas flow is negligible due to the low heat capacity and thermal conductivity of the gas, as can be verified by an elementary calculation. Experimental thermal shock data will be presented in the next section.

## 4. LABORATORY EXPERIMENTS

### 4.1 Snowflake morphology

When liquid CO<sub>2</sub> at 750 psi is throttled through a nozzle and expands into a volume at 1 atm pressure, the liquid

undergoes a phase change to the solid state<sup>10</sup> The shape and size of the resulting snowflakes depend on the details of construction of the nozzle as well as the local conditions under which the flake formed in the gas stream If the liquid escapes through a needle valve, flake diameters are generally very small (approximately 10 microns), whereas escape of the liquid through an aperture 1 mm across leads to flakes about 1 mm in diameter Crystallographically, dry ice has a pyrite-like underlying crystal structure which is a cubic close packing of CO<sub>2</sub> molecules centered in their carbon atoms<sup>11</sup> Such crystals have a bulk habit which is frequently cubic, but crystals may also form octahedrons or other complex polyhedrons<sup>12</sup> Octahedrons showing unequal development of their eight faces may resemble thick hexagonal plates with alternating long and short sides<sup>12</sup> Snowflakes may be either individual crystals or low density graupel-like collections of crystallites Because of the short lifetime of CO<sub>2</sub> snowflakes it was not possible to obtain direct photographs of them However, it was possible to photograph the footprints left by these flakes as they collided with a clear acrylic resin dissolved in a mixture of aromatic solvents and ketones The coating was sprayed onto a microscope slide which was immersed in the stream of flakes Drying was very rapid as the fast moving stream of CO<sub>2</sub> carrier gas rapidly removed solvent vapors, thereby "freezing" the footprints in the coating before spreading could obliterate them Fig's 5 and 6 are examples of results obtained by the technique above. Footprints left by large flakes could be recorded by ordinary optical microscopy, but the footprints left by small (10 micron) flakes had to be examined by scanning electron microscopy (SEM) SEM examination requires the acrylic samples to be coated with 300 Å of gold-palladium alloy Producing good imagery from such specimens is extremely difficult since the electron beam is capable of melting the acrylic substrate

#### 4.2 Cleaning efficiency

In this section the percentage of particles removed by CO<sub>2</sub> cleaning under various conditions is presented Clean mirrors were produced by sputtering 300 Å ( $\pm$  30 Å) of gold-palladium alloy at a rate of 2.5 Å/sec onto soda-lime glass substrates in a residual argon atmosphere at 10<sup>-6</sup> Torr The substrates were ultrasonically cleaned, prior to deposition, in distilled deionized water and then blown dry with bone dry nitrogen before deposition After the mirrors were finished, they were contaminated with an NBS abrasive standard known as "fine Arizona road dust" to a contamination level of 7 ( $\pm$  3) particles per 1000 square microns (Fig 7) The particle size distribution of Arizona road dust is shown in Fig 8 After cleaning a 1 cm<sup>2</sup> test sample for 1 sec with 10 micron diameter flakes, no particles remained anywhere on the sample that could be detected with either the optical or electron microscope However, some particles remained after cleaning a sample with large 1 mm diameter flakes for 5 sec In the latter case the residual contamination did not exceed 1 particle per 1000 sq microns One exceptionally large elliptically shaped particle (semimajor axis = 3 microns) is shown in Fig. 9 However, most residual particles left behind after cleaning with large flakes are submicron-sized and cannot be seen with an optical microscope at visible wavelengths

The cleaning efficiency as a function of contaminant composition was also studied Many microscopic contaminants at the MMT are like those in Fig 1 (i.e., crystalline mineral particles) Some of these crystals show a termination, under the electron microscope, whose apex angle has been measured and, in many cases, matches the angle between the r faces of a termination of crystalline quartz<sup>13</sup> The rocks on top of Mt Hopkins are fairly well consolidated; however, there is a significant amount of friable decomposed granite which is used as landscaping material around the observatory, and it is suspected that this is the source of at least some of the MMT's contaminants In the future it might be wise for observatories to use a more solid landscaping material like "river rocks." The previously described cleaning tests with Arizona road dust are a good model for the type of cleaning required at the MMT, since Arizona road dust is rich in silica

Next, CO<sub>2</sub> cleaning equipment was moved to Mauna Kea, Hawaii, for cleaning experiments at the Canada-France-Hawaii Telescope (CFHT) D Salmon of CFHT assisted On Mauna Kea one of the major contaminants is a fine basaltic volcanic ash Fig's 10a and 10b show one of the CFHT's Coude mirrors before and after cleaning with large flakes for 20 seconds Only two chemisorbed contaminants near the edge of the mirror failed to be removed In short, CO<sub>2</sub> snow was able to remove 97.7% of the contaminating particles on the CFHT's mirror

At Lick observatory on Mt Hamilton, California, the microcontamination problem involves removal of optically black particles from mirrors and other optics Chemical analysis showed these particles to be carbonaceous, and it was, at first, thought that these particles were specks of optically black paint from telescope structural components. However, it now appears that these particles are small pieces of soot which are generated by annual summer grass fires in the surrounding area This latter conclusion is supported by the finding of similar small particles on outside windows of observatory buildings and on vehicles parked outside Complicating the

problem is the high winter relative humidity, which may frequently exceed 80%, thus resulting in condensation during the cooler parts of the 24 hour day. Such condensation may encourage chemisorption through chemical reactions between the soot and the aluminum since some soot particles show a tiny corrosion ring around them at a linear magnification of 3x.

The 24 inch primary mirror of the auxiliary Coudé telescope at Lick was aluminized on Nov. 26, 1986 and had never been cleaned. On Feb. 8, 1989 M. Owens of the Lick observatory staff removed the mirror from its cell for cleaning with CO<sub>2</sub> snow. Reflectometer measurements on the mirror were made by R. Laub and B. Brown of the Lick observatory staff before and after cleaning. Before cleaning the reflectivity of the mirror in blue light (wavelength approx. 4500 Å) was  $86\% \pm 0.6\%$  of the Lick observatory standard mirror. In red light (wavelength approx. 6500 Å) the reflectivity was  $91.5\% \pm 0.8\%$ . After CO<sub>2</sub> snow cleaning with small flakes the reflectivity rose to  $91.8\% \pm 0.3\%$  of standard in blue light and  $99\% \pm 0.5\%$  in red light. All the larger black particles were removed, but careful examination of the mirror with a 3x jeweler's loop reveals that some of the very smallest black particles remained, accounting for the remaining small loss in reflectivity. Solution washing brought the mirror back to 100% of standard in both blue and red light. A second test compared the cleaning efficiency of large and small flakes on an uncleaned portion of the 24 inch mirror. The reflectivity in red light after cleaning with large flakes was  $99.3\% \pm 0.5\%$ . This final reflectivity, however, is not considered to be statistically different from that achieved with small flakes. A third, and final, test was conducted on an optical flat. The flat was cleaned with a pulsed high pressure jet of dry nitrogen (pressure approx. 2000 psi). This cleaning resulted in a final reflectivity of  $93\% \pm 0.8\%$  in blue light. Next, the same mirror was cleaned with CO<sub>2</sub> snow. This second cleaning caused the reflectivity to rise again to  $95.5\% \pm 0.4\%$  in blue light.

#### 4.3 Scratching studies

A critical question which must be answered is whether a snowflake, or the dust which it moves on collision, are capable of scratching a solid mirror surface. To answer this question, gold-palladium alloy films like those previously described were contaminated with NBS fine Arizona road dust. After cleaning with both large (1 mm) and small (10 micron) flakes, the surfaces were examined for scratches which might have been made by flakes or skating particles. No evidence of scratching was found. Any surface modification (defects) induced by CO<sub>2</sub> snowflake cleaning must have dimensions smaller than 0.05 microns, the limit of resolution of the surface inspection performed. Fig. 9 shows a typical gold-palladium coated surface after cleaning.

#### 4.4 Contamination deposition

One problem with CO<sub>2</sub> snow cleaning is that there is some organic/oil contamination in some of the liquid CO<sub>2</sub>. This contamination arises from the fact that liquid CO<sub>2</sub> is a good solvent for oils and readily dissolves contaminants such as compressor lubricants or contamination from the walls of the tanks used to store the liquid CO<sub>2</sub>. Consequently, a detailed study has been made of organic contaminant concentration and identification as a function of CO<sub>2</sub> purity. First, industrial grade CO<sub>2</sub> was examined for impurities. Typically, such gases are 99.5% to 99.9% pure (by volume). The few tenths of a percent of impurities include, in addition to atmospheric gases, some very troublesome organics which can affect performance of optics and electronic parts cleaned with CO<sub>2</sub> snow. The contamination may even be visible to the naked eye on certain substrates and under suitable lighting conditions. Infrared absorption studies of the residue rinsed from glass surfaces by spectrally pure freon TF revealed that structurally these impurities have the form of poly(1-pentyltetramethylene). Such impurities occur at a level of about 39 ppm by volume (Fig. 11). If the grade of gas is further improved by fractional distillation to 99.99% (medical grade), then heavy polymers are removed and lighter complex molecular compounds like dimethylsiloxane and decylolate dominate the organic impurity mix (Fig. 12) at the level of about 7 ppm. With this grade of gas, contamination can occasionally be detected microscopically as a few isolated beads of organics (approx. 1 micron across) per square decimeter. When spectral grade CO<sub>2</sub> (99.9999% pure by vol.) was examined for organic content, little was observed, except one small peak just barely above the noise at extreme absorbance scale magnification (Fig. 13). There was insufficient data to determine the contaminant's structure in any detail, but the peak in question is due to the presence of a carbonyl group. Furthermore, switching to an ultrapure hexane carrier removed the carbonyl peak but added other "noise" peaks. From this it was concluded that the carbonyl peak was due to the freon TF carrier, and that the organic impurity level of the spectral grade gas is less than about 110 parts per billion.

In addition to spectral examination of residues, a stiction-friction test on a super-smooth evaporated carbon film was conducted. This film had an rms roughness of only 30 Å. The coefficient of friction of the film surface was

measured to be 0.25 to 0.3 even after prolonged spraying with spectral grade CO<sub>2</sub>. These tests had to be conducted in a clean room area.

#### 4.5 Thermal shock

Generally speaking, thermal shock is not a problem during CO<sub>2</sub> snow cleaning because of the short time required for cleaning and the gaseous boundary layer preventing contact between the solid CO<sub>2</sub> and the surface. No film cracking or peeling of a reflective coating due to local chilling has ever been observed as a consequence of CO<sub>2</sub> snow cleaning. The reason for this can be understood in terms of Fig. 14. A 2 mm thick aluminum plate was bombarded at a fixed point with CO<sub>2</sub> snowflakes for 1.5 minutes, at a range of 15 cm from the nozzle, and with a CO<sub>2</sub> flow of 0.9 l/sec (at STP). The flakes struck the surface at an angle of 60° to the normal and a chromel-alumel thermocouple attached to the back of the plate, just under the point of impact, recorded the temperature of the plate. The temperature fell at an almost linear rate of 3-6°C every 15 seconds. Typically, cleaning requires about 1 to 5 seconds per square decimeter, depending on how adhesive the contamination is. Therefore, it is reasonable to expect temperature drops of 0.2 to 2.0°C during normal cleaning operations. Such temperature changes are about 1000 times too small to produce cracking of a reflective coating.<sup>14</sup>

Condensation is not normally a problem during CO<sub>2</sub> cleaning because of the small temperature drops involved and the fact that the bone dry CO<sub>2</sub> acts as a "shield gas" to keep moist air away from the surface to be cleaned. However, when surfaces are excessively chilled by long dwell times, as in the experiment described here, the end of CO<sub>2</sub> cleaning may signal the initiation of condensation. The condensate may be in the form of small water droplets, or in the case of very cold surfaces, frost. Such condensed moisture is not necessarily clean and may contain particulate seeding nuclei from the moist air layer over the cold surface.

#### 5. CONCLUSION

Clearly, the CO<sub>2</sub> snowflake cleaning method has many advantages over solution cleaning for routine maintenance. The method is fast, easy to use, gentle, and effective. There is, however, one other bonus; it is inexpensive. Fig. 15 summarizes the economics of the situation. The cost of liquid CO<sub>2</sub> rises more or less linearly with the number of "9's" of purity. For astronomical/optical purposes gas of four 9's purity (99.99% pure) is adequate. Any inadvertent deposition of organics by this gas would be so small that the mirror would have to be washed because of chemisorbed dirt long before the organic buildup could ever be an important obscurant. However, other applications might demand the higher grades of gas.

A future paper will describe practical applications and methods in more detail.

#### 6. ACKNOWLEDGMENTS

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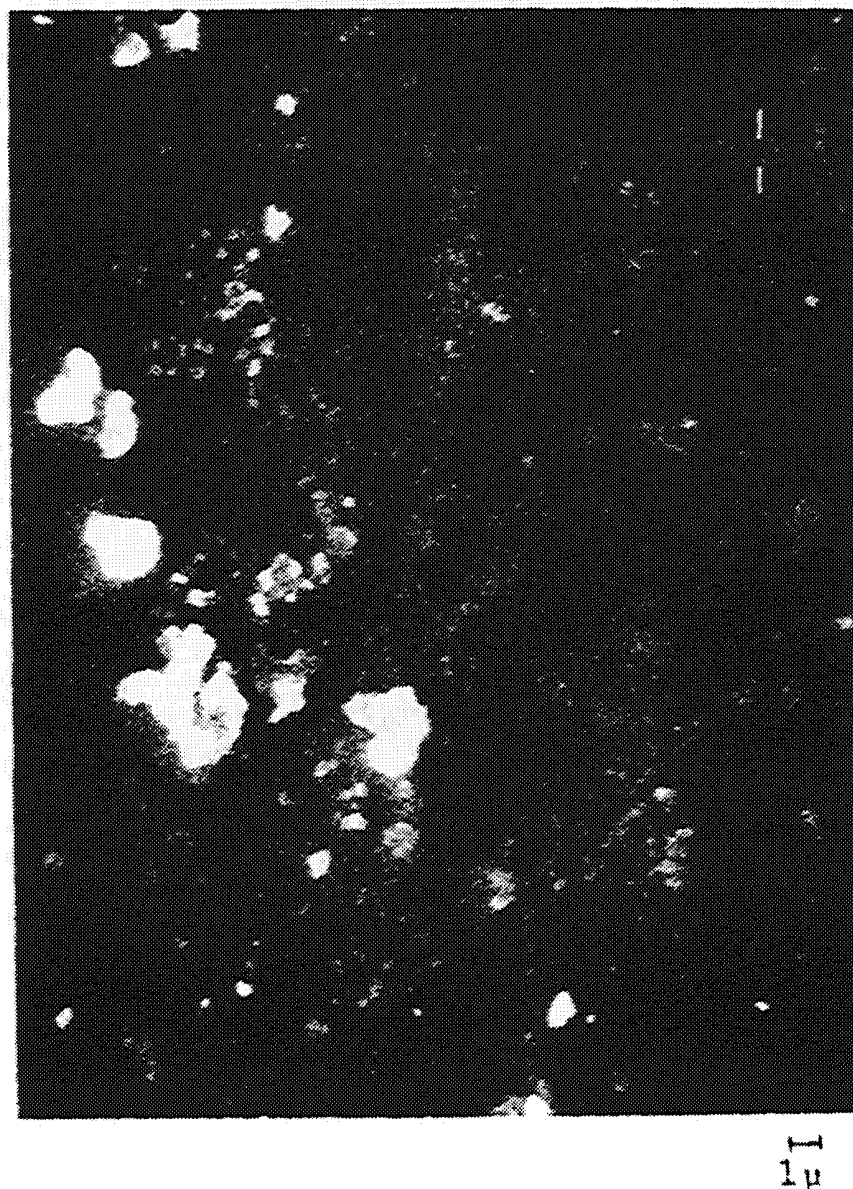


Fig 1 SEM micrograph of contamination on a microscope slide-sized test aluminum mirror attached to the mounting of the MMT from Aug 15, 1984 to Apr 2, 1988. The test mirror pointed in the same direction as the MMT primaries and therefore provided a small convenient monitor for assessing the accumulated dirt. Pointed terminations can be seen on a few crystals of debris.



Fig 2 Macroscopic mirror debris collected from the MMT These objects include a large number of insects (especially moths and ladybugs), hair, leaves and stems from surrounding vegetation, and light man-made materials such as styrofoam



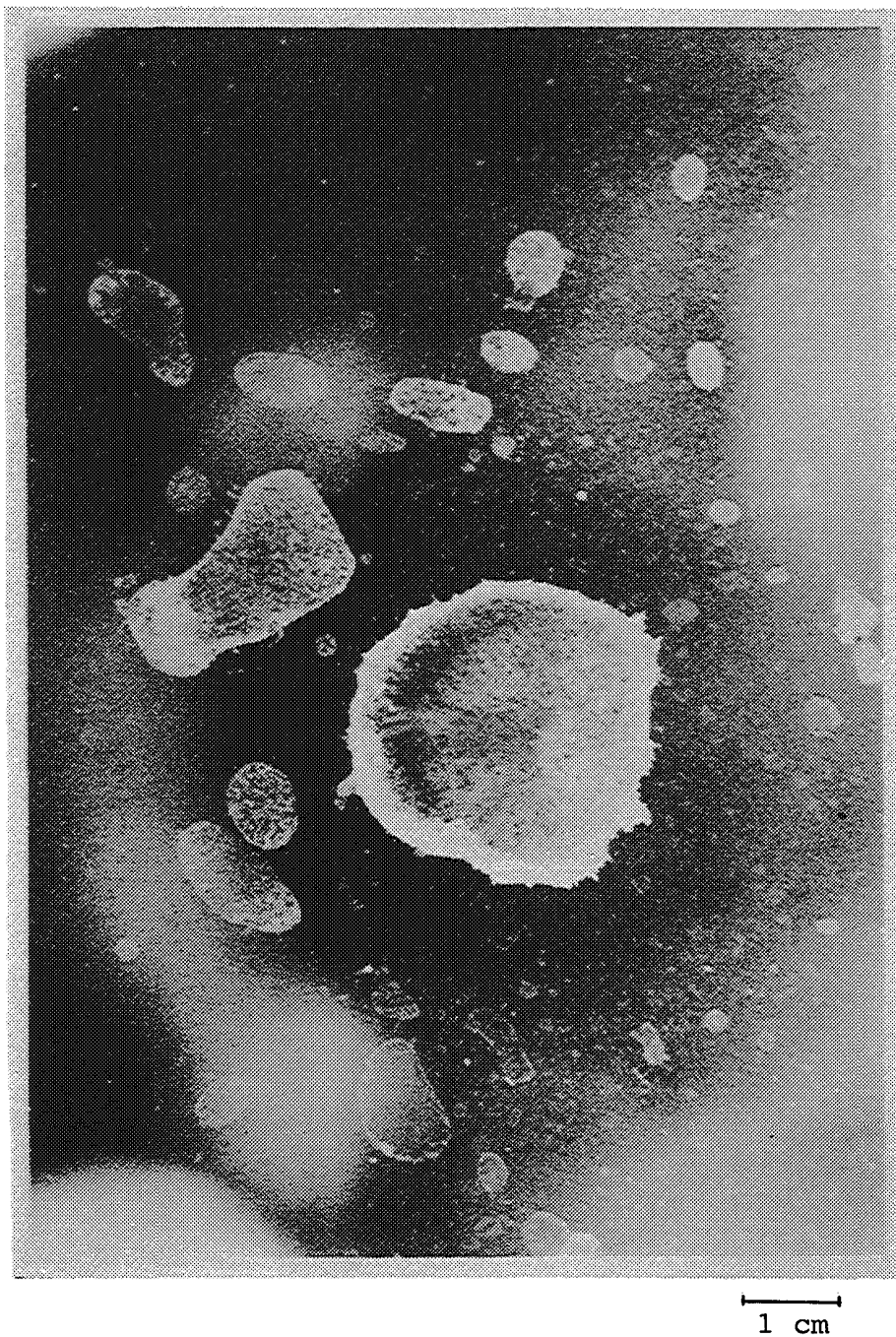


Fig 3 "Water Spots" left behind on one of the MMT's primary mirrors by a condensation drop from the dome structure. Such drops are rich in dissolved minerals from dust particles. After evaporation of a drop these minerals are redeposited on the mirror. In the photo above, liquid drained to the right (toward the lowest part of the mirror) during evaporation. Several satellite stains were created around the main deposit by splashing.





Fig 4 A small severe local corrosion spot on an aluminum test mirror attached to the mount of the MMT on Aug 15, 1984 and removed on Apr 2, 1988 In addition to the corrosion spot, a large amount of particulate debris can be seen residing on the surface



Fig 5 The footprint of a single crystal of CO<sub>2</sub>

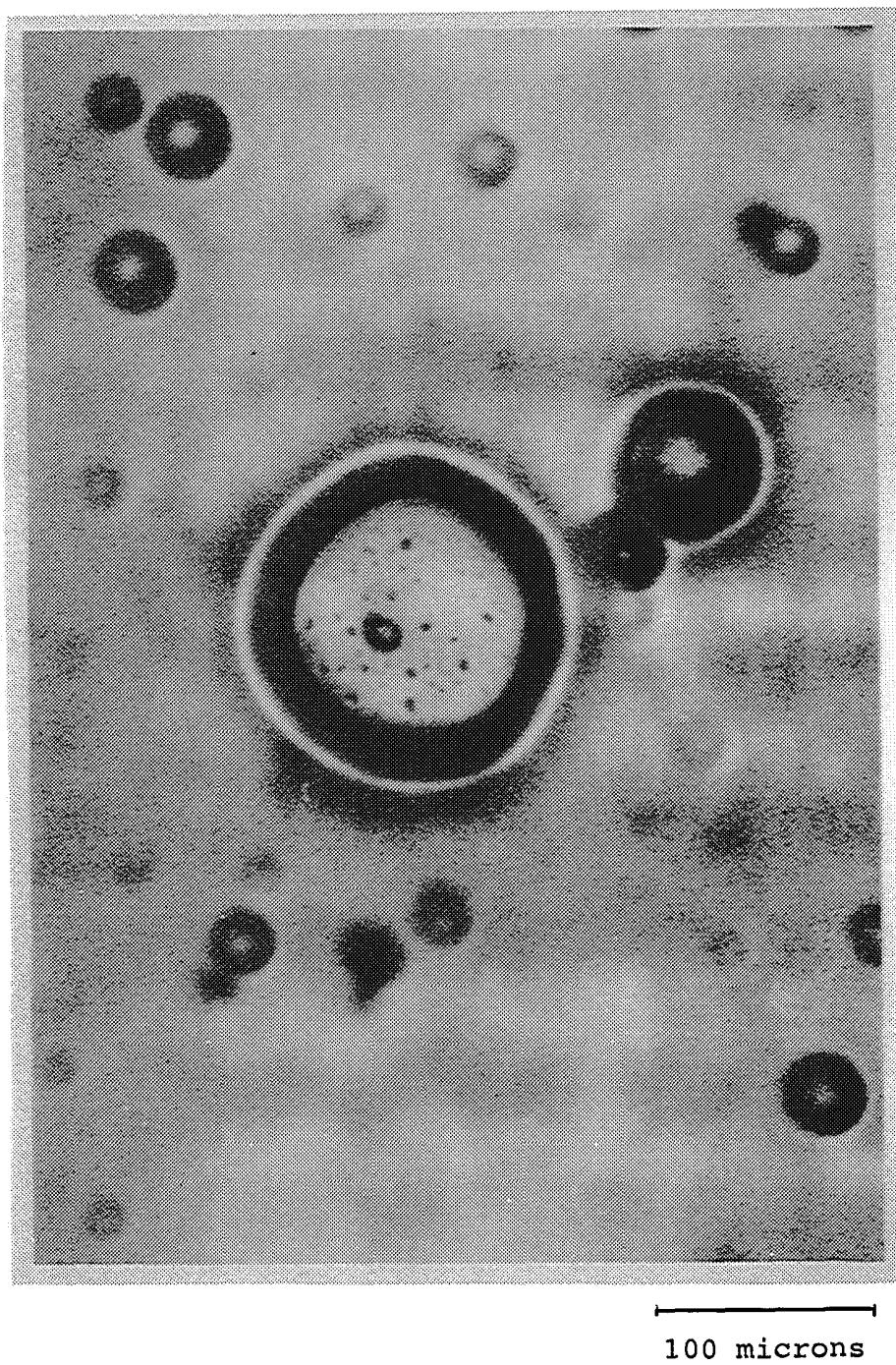


Fig 6. A graupel-like CO<sub>2</sub> "snowball" has struck the soft acrylic leaving a crater. At the bottom of the crater impressions can be seen from individual CO<sub>2</sub> crystals. One of these impressions is distinctly triangular and represents penetration of a corner of a cubic crystal into the plastic.



Fig 7 A 300 Å thick film of gold-palladium alloy contaminated with fine Arizona road dust



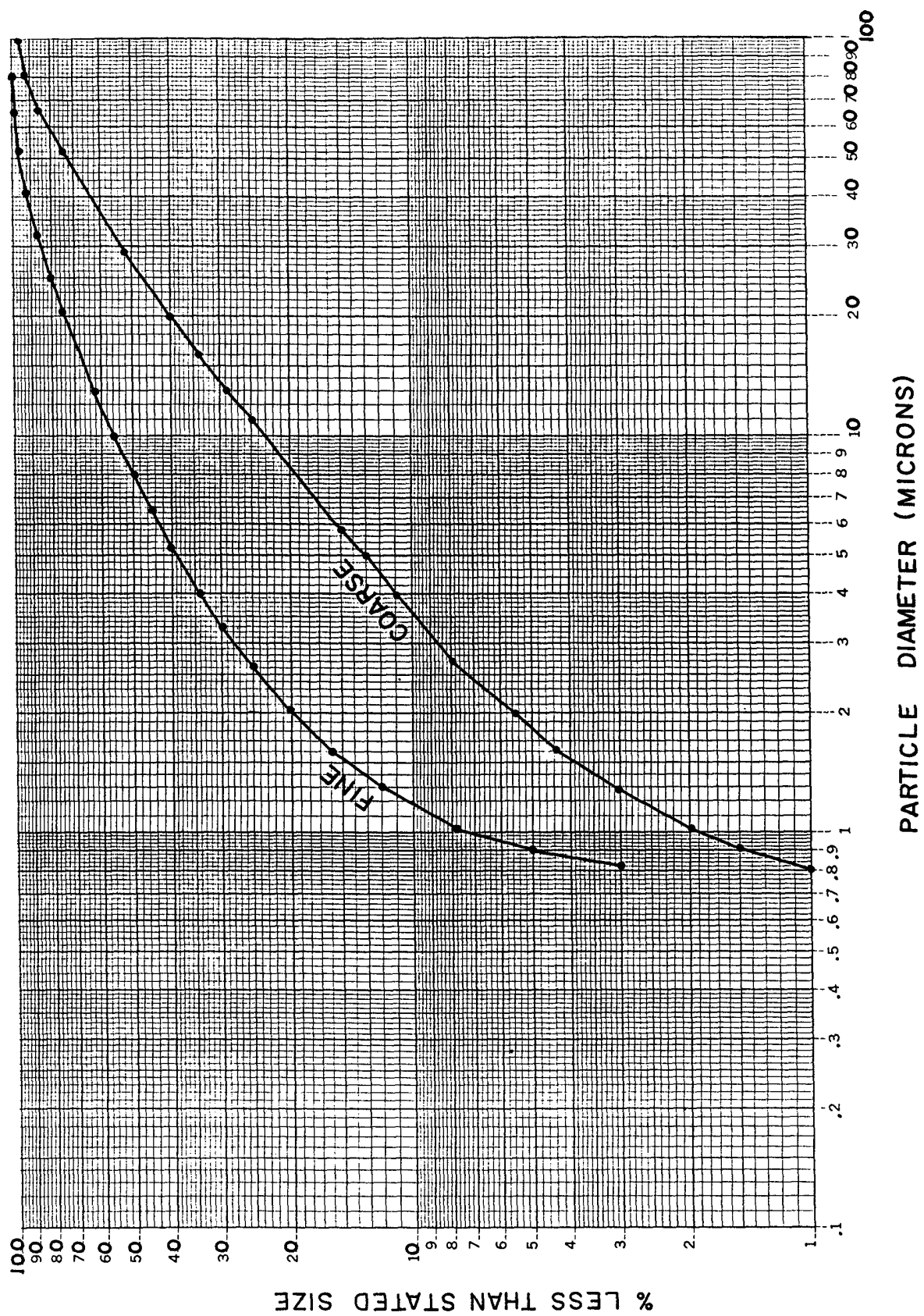
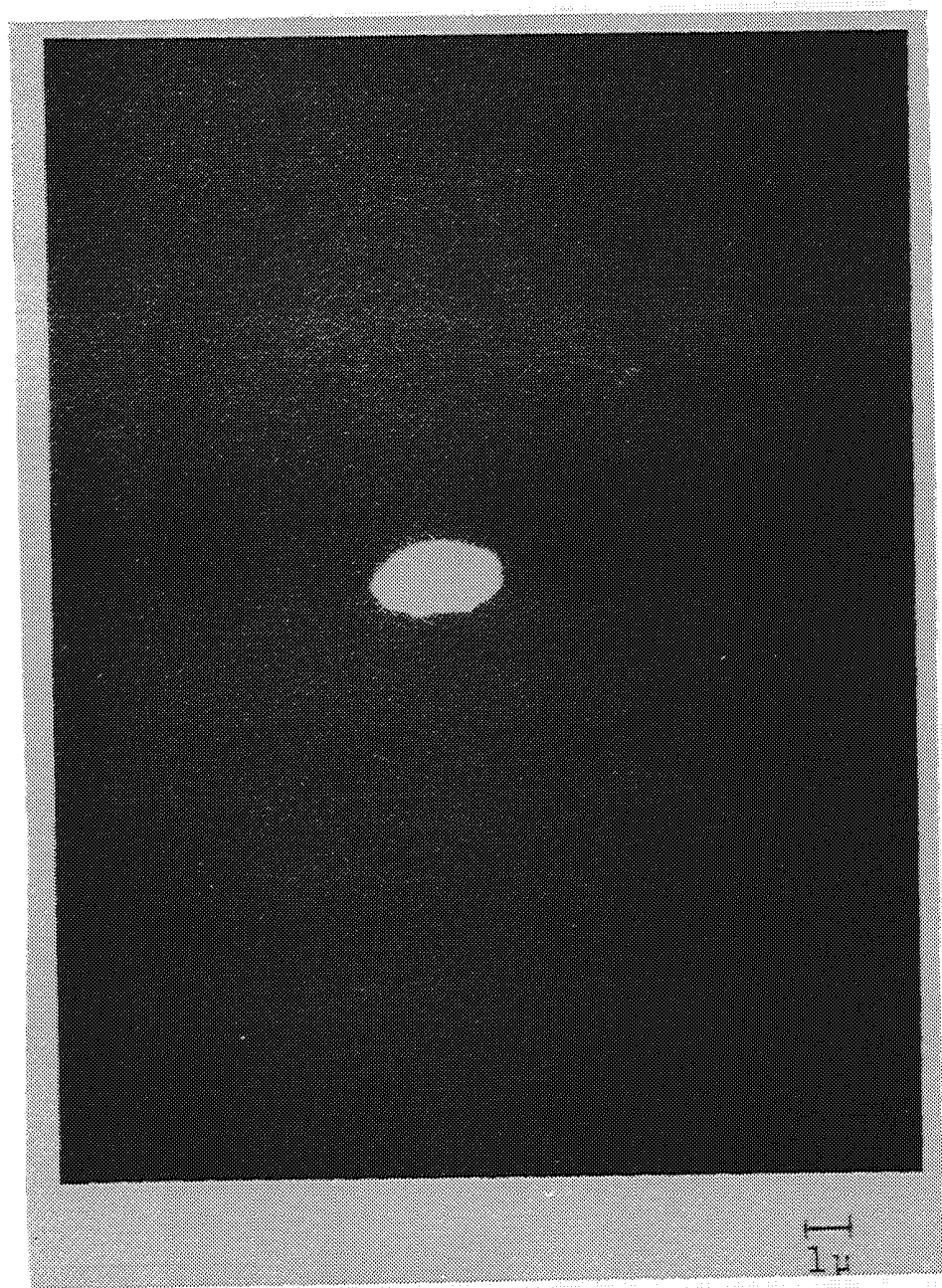
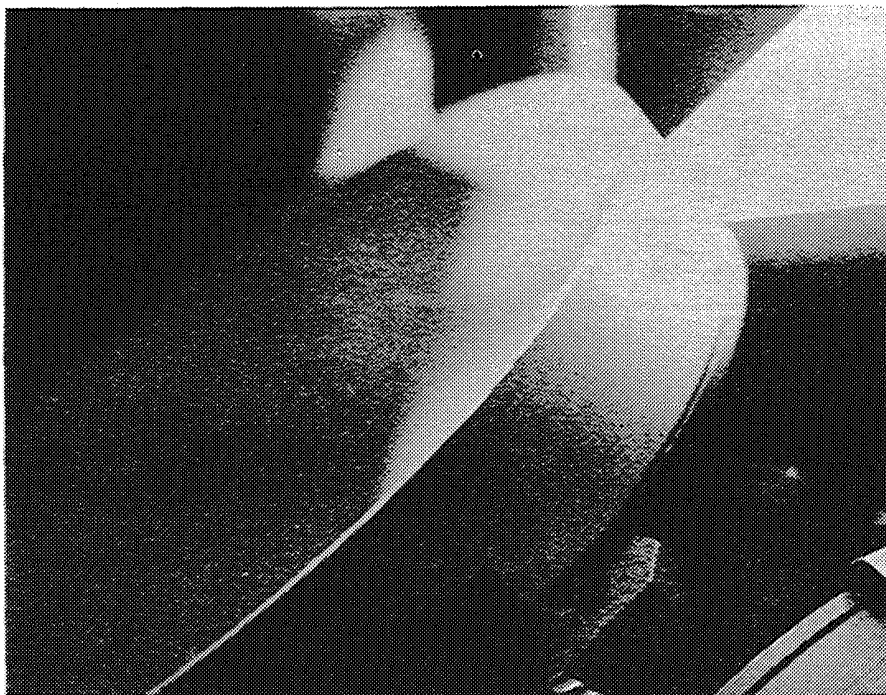


Fig. 8. Particle size distribution for fine and coarse Arizona road dust.

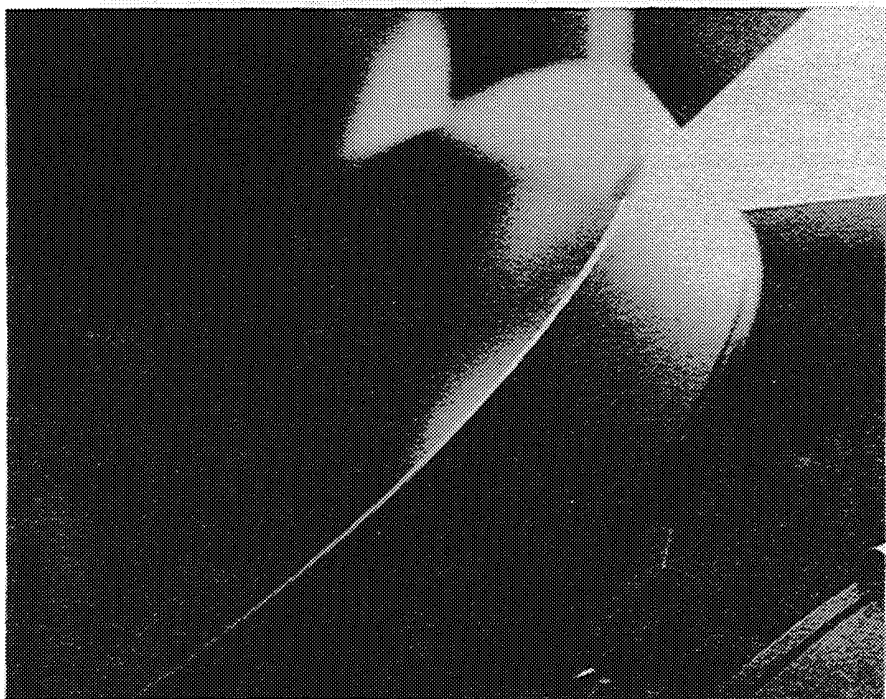


**Fig 9** Contaminated gold-palladium alloy film after cleaning with large (approx 1mm diameter) flakes. A few particles still remain, but their number density has been reduced by almost an order of magnitude. No evidence of scratching by either snowflakes or skating dust particles can be seen.





A



B

Fig 10 A 12" Coude mirror at the CFHT before (A) and after (B) a 20 second cleaning with large CO<sub>2</sub> snowflakes. Grazing incidence illumination was used to reveal the remaining particles. It was not possible to remove two small chemisorbed particles which serve as useful fiducials in these photos.

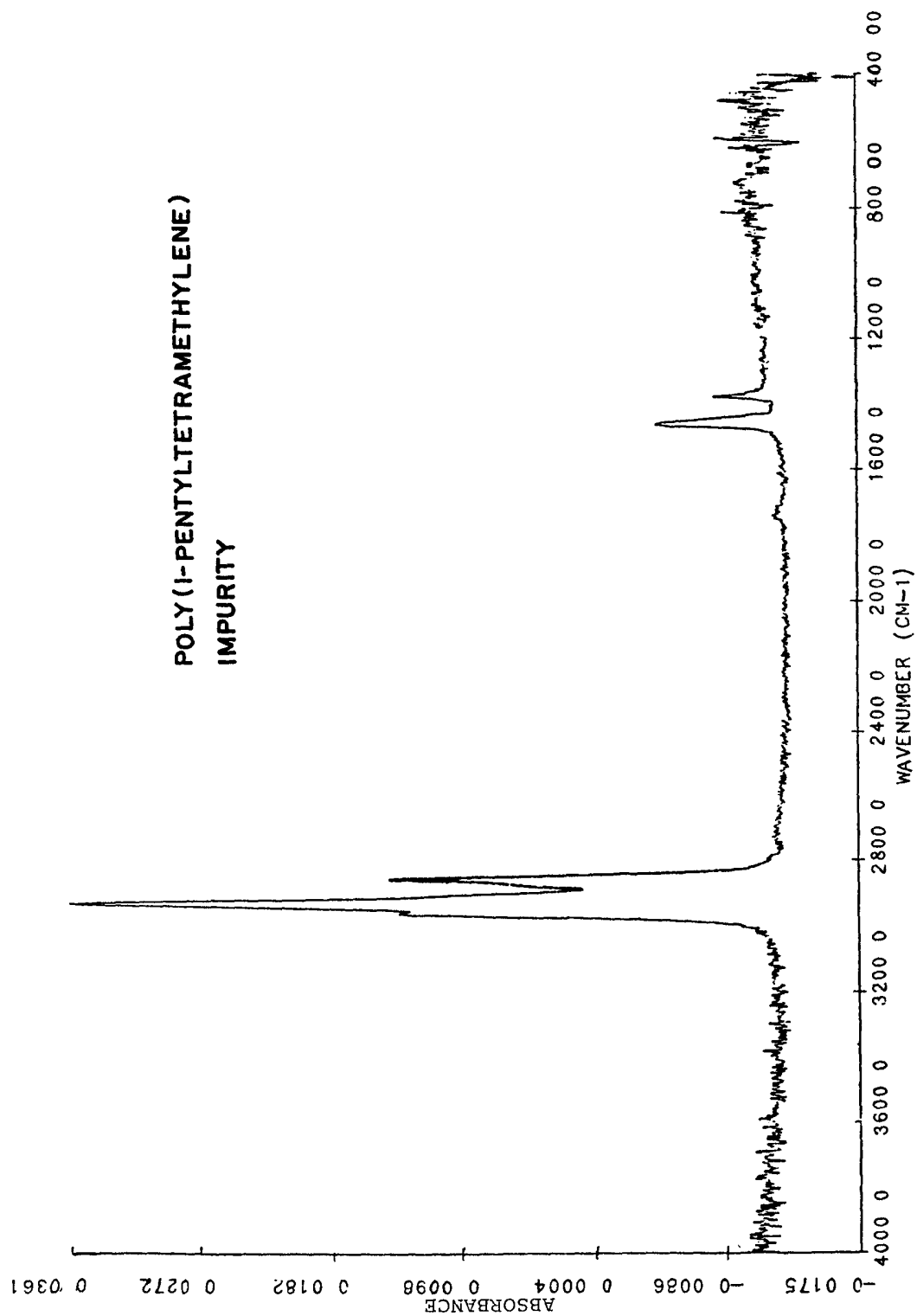


Fig. 11. Infrared absorption spectra for industrial grade CO<sub>2</sub>. The principal impurity is poly (1-pentyltetramethylene).

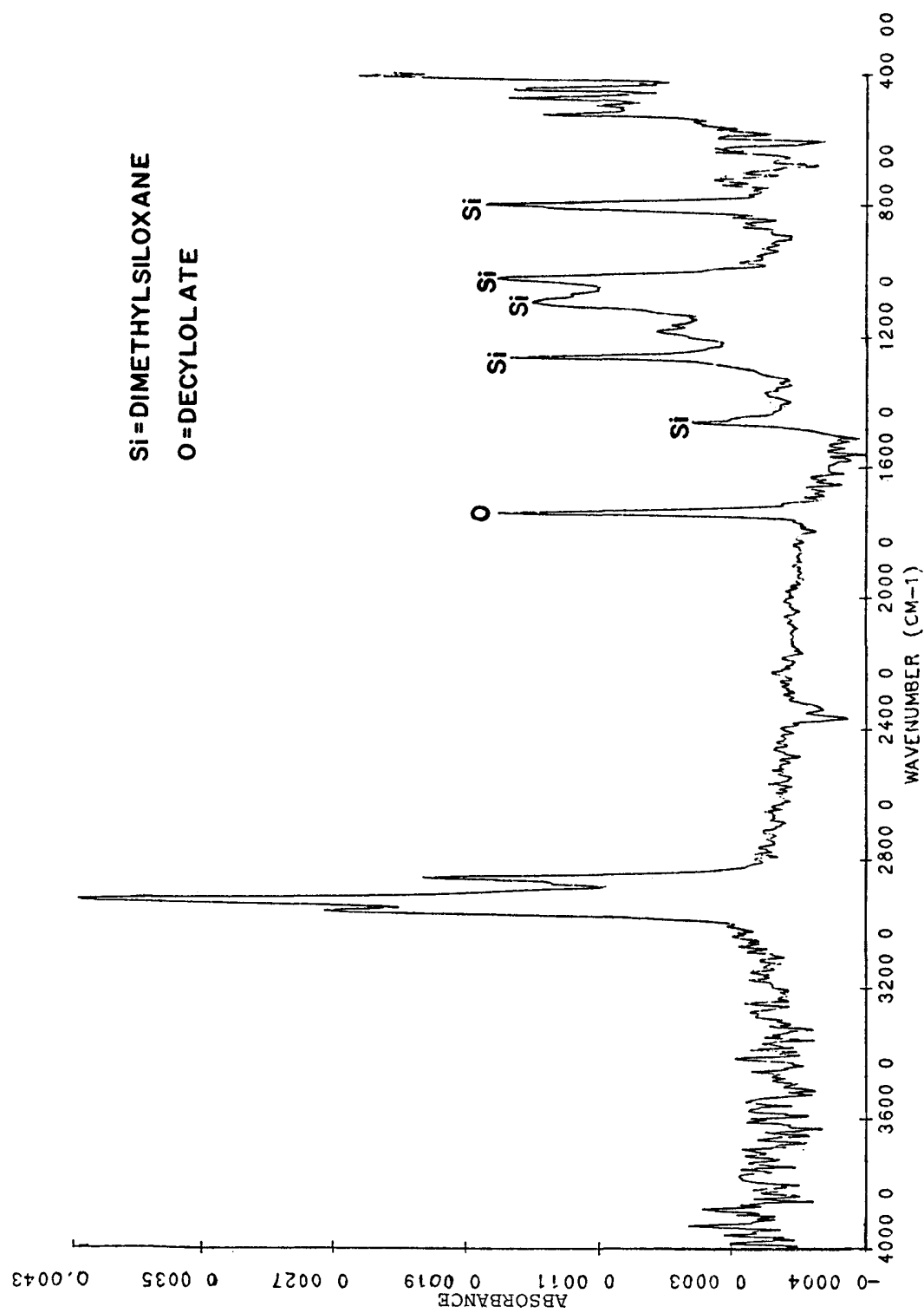


Fig. 12. Infrared absorption spectra for a high-quality medical grade of CO<sub>2</sub>. Here the principal impurities are dimethylsiloxane and decylolate.

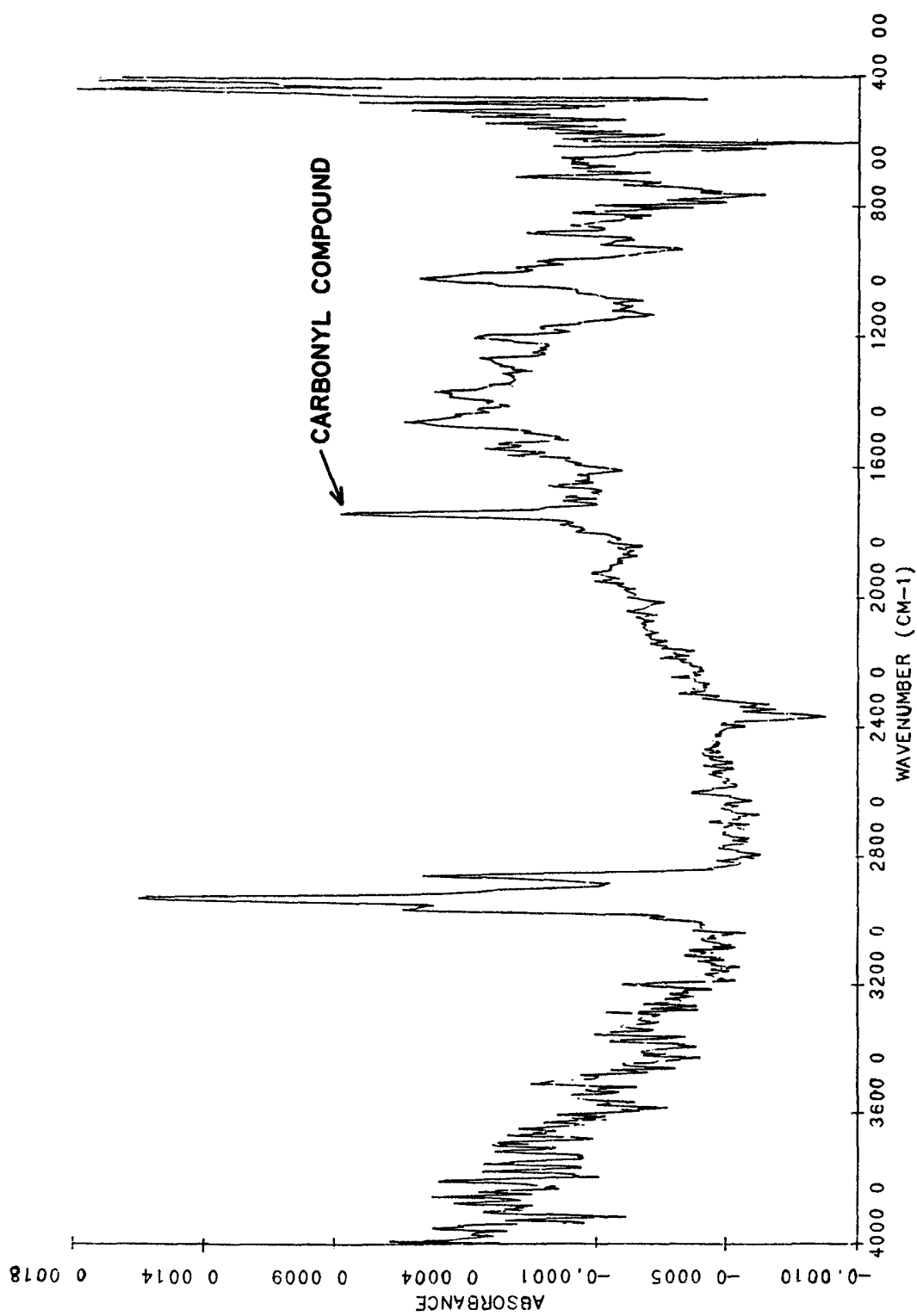


Fig. 13. Infrared absorption spectra for spectral grade CO<sub>2</sub>. A carbonyl impurity appears to be present, but this impurity was later ascribed to the freon TF carrier.

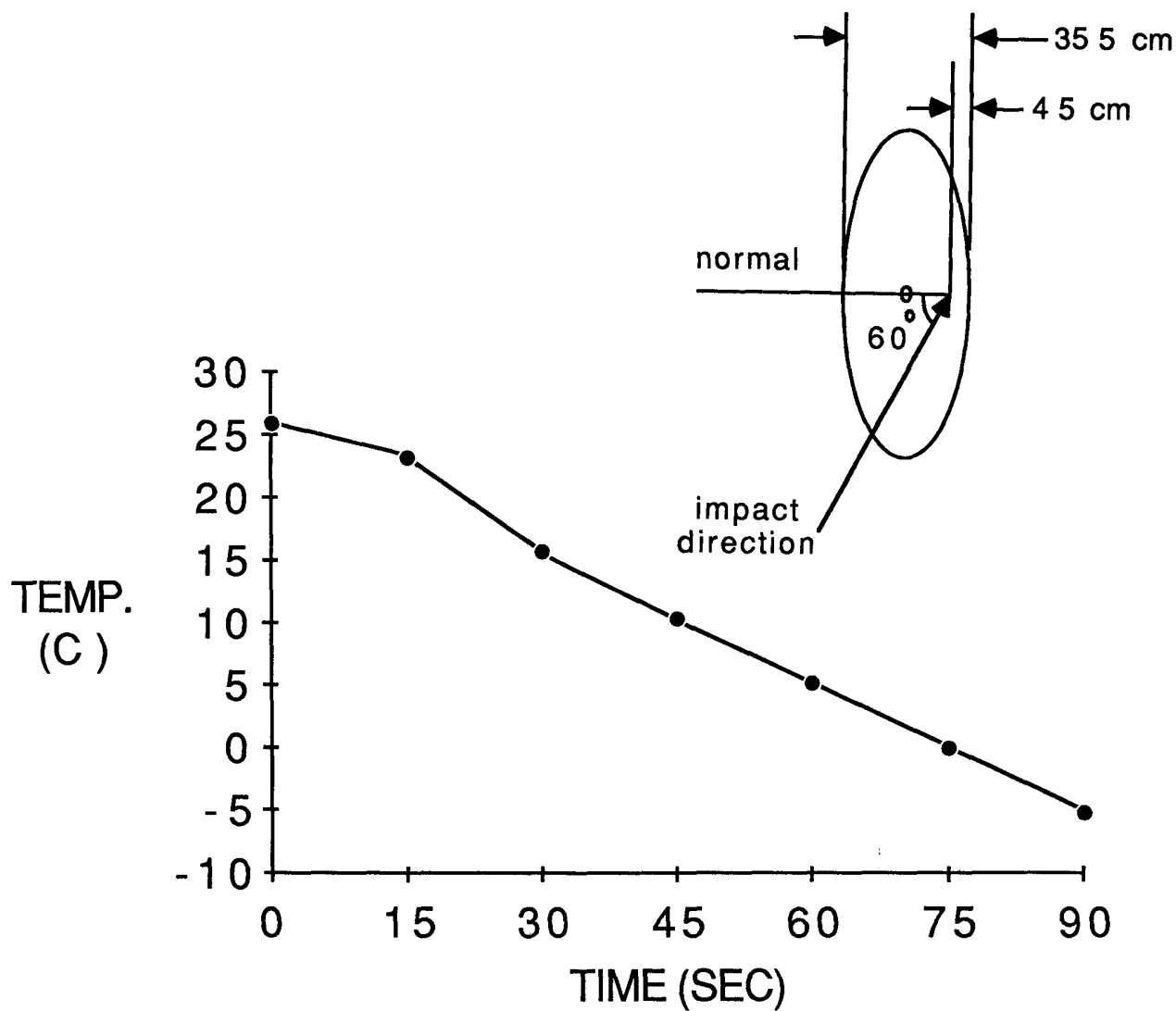


Fig 14 The change in temperature as a function of time for a 2 mm thick Al plate sprayed with small CO<sub>2</sub> snowflakes at a range of 15 cm and a flow of 0.9 l (STP)/sec. The plate was mounted to a steel support at its center. Temperature drops were typically 3-6°C for every 15 sec of cleaning. Error bars of  $\pm 2^{\circ}\text{C}$  should be associated with each data point.

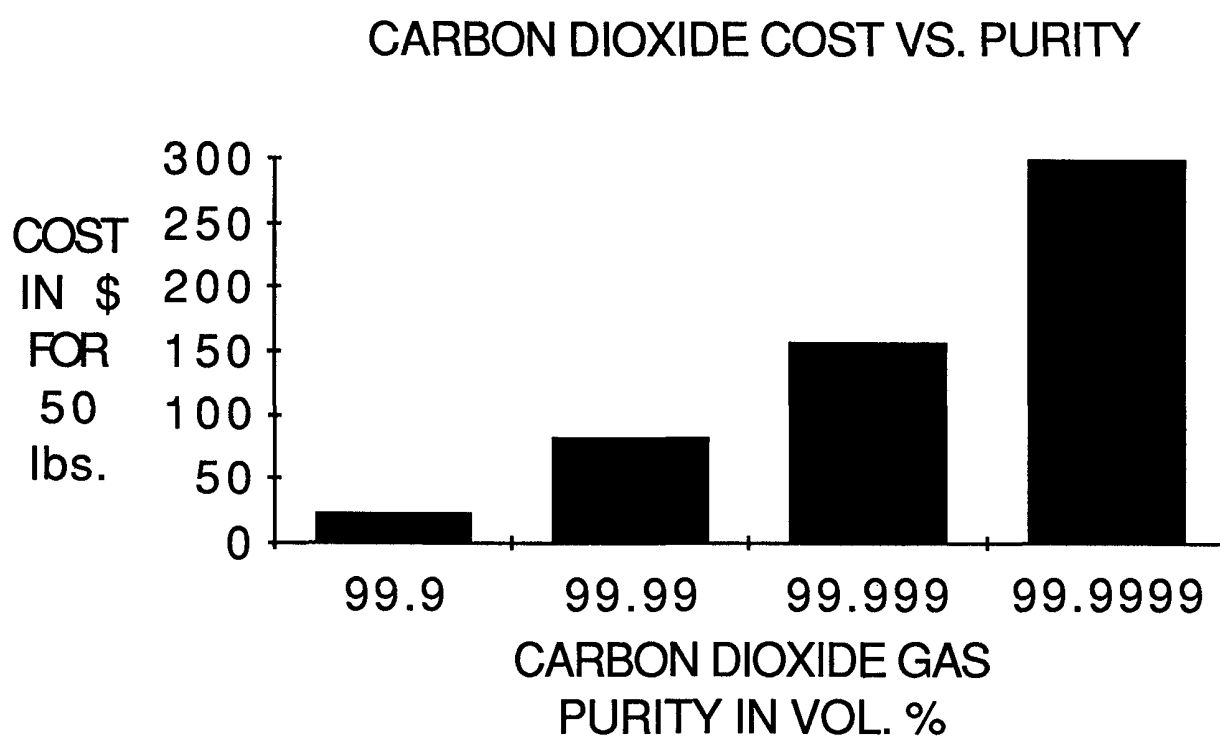


Fig 15 Cost versus CO<sub>2</sub> purity for 50 lbs of gas The cost of the 99 999% pure gas is an exception and represents the price of 100 l of gas at STP