

## RAPID COMMUNICATION

# Calorimetric radar absorptivity measurement using a microwave oven

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**Abstract.** The measurement of the microwave absorptivity of materials typically requires expensive and arcane equipment and methods. I show that, for moderately absorbing materials (loss tangent  $\delta \simeq 0.005$  or higher), useful measurements can be easily made using a thermometer and a normal domestic microwave oven: the heat absorbed for suitably designed sample size and geometry relates directly to the absorptivity. This technique, although not supremely accurate, is quick and inexpensive and may be useful for rapid investigations, educational projects and situations in which samples are awkward to handle. I show that water ice spiked with even only 0.2% ammonia is about three orders of magnitude more absorbing than is pure ice at  $-80^\circ\text{C}$ .

**Keywords:** microwave, dielectric constant, absorptivity, ices

## 1. Introduction: measurement of microwave absorptivity

The strength of a radar echo from a planetary surface depends on the scattering properties of the surface itself, the density of scattering surfaces beneath the surface and the absorptivity of the subsurface medium. The latter property is of particular interest for arid regions on Earth, or very cold ice-rich surfaces, where the absorptivity is low and radar energy can penetrate deeply.

Methods for measuring the microwave absorptivity of materials are summarized by Von Hippel [1]. Typically a calibrated microwave source and receiver are needed and the sample is placed in a waveguide or cavity. The absorptivity (and also in many cases the real part of the complex dielectric constant) is inferred from the reduction in magnitude of the measured signal.

In this paper, I show that the absorptivity can be related to the temperature rise of a sample immersed in a powerful microwave field. These measurements were motivated by an interest in the microwave properties of cryogenic ices and, in particular, the hint from lower-frequency measurements that ammonia-doped ice might be strongly absorbing. This has important implications for the interpretation of radar data to be acquired from the moons of Saturn, Titan in particular, by the Cassini spacecraft launched in 1997.

## 2. Heating in a microwave oven

The heating of materials in a microwave oven is a complicated process [2]: the radiation field within the oven is non-uniform and is perturbed by the presence of materials within the cavity, which both absorb and reflect the radiation. Within the material sample itself, the radiation that is not reflected is refracted at the edge of the sample, leading to focusing of radiation. The absorptivity of the material also can lead to non-uniform heating—the absorption depth in pure water, for example is around 2 cm, whereas for salty water (a much more lossy dielectric) the absorption length is only 0.7 cm, leading to substantial edge heating [3].

In an ideal oven, with no absorbing material except the sample, the microwave power absorbed by the sample would be independent of the sample properties and would equal the power emitted by the magnetron. Any power not absorbed by the sample initially would rattle around within the cavity until it was absorbed.

Thus, to force the power absorbed by the sample to relate to its absorptivity, the microwave power should pass on average only once through the sample. One possibility would be to have an open magnetron, such that any power not absorbed by the sample simply disappears to infinity. This approach offers conceptual and analytical simplicity, although it presents laboratory-safety concerns and requires irreversible modification of the oven.

Another approach would be to place the sample inside a container with partially absorbing walls, such that radiation that enters the container once will typically not survive leaving and re-entering it and so can pass through the sample only once or twice. A two-walled container, with around 2 cm of water between the walls, corresponds to the required optical depth. This geometry was tried, with some success; but it has been found after extensive experimentation that an adequate approximation to this geometry is simply to put the sample in the centre of the oven and a large beaker of water in one corner.

Another important factor is the size of the sample. If a sample's characteristic dimension is large compared with the absorption length, then all of the radiation entering the sample will be absorbed, again defeating the measurement. If, on the other hand, the optical depth of the sample is small, that which is absorbed depends on the optical depth and thus on the absorptivity.

### 3. Practical considerations

Assuming a sample size and microwave field that relate the power absorbed by the sample to its absorptivity, the next consideration is how to quantify the absorbed power. In a thermally isolated sample, this is linearly related to the density and specific heat capacity of the sample. Measuring the temperature change of the sample after a known duration of heating should therefore allow the energy absorbed to be estimated.

For a real sample conductive heat transfer to and from the sample can be minimized by surrounding the sample by a non-conductive but microwave-transparent material like polystyrene foam. A major difficulty for liquids with significant vapour pressures (including water and other solvents that might be used for calibration) is eliminating evaporation losses. This can be a particular problem for strongly absorbing samples (and led to early underestimates of the absorptivity of saline solutions—the strong edge heating raised local temperatures and increased evaporation losses [3]).

Heat leaks to or from the sample can be minimized by making the sample as large as possible, since the heating in an optically thin sample is uniform and therefore proportional to its volume, but losses relate to the surface area. However, unless temperature measurements are made throughout the sample, the assumption that the sample is isothermal has to be made, so the sample should be small to allow uneven heating (by focusing effects, for example) to be averaged out.

For an accurate temperature-change measurement, a small sample presents some difficulties (since the sample shares its heat with the thermometer). Furthermore, a larger temperature change is easier to measure, but leads to higher losses from the sample and possibly a change of absorptivity (for example, the loss tangent of water decreases from 0.25 to 0.05 between 3 °C and 80 °C [4]). For a sample with entirely unknown absorption, a large sample should be used first and the sample size should be reduced thereafter if appropriate.

It would be most convenient and instructive to measure the temperature of the sample continuously and *in situ*. However, this leads to electrical sensors like thermocouples

act as antennae and cause local heating within the sample [5]. This problem can be avoided by using the non-conducting fibre-optic sensors (using temperature-dependent fluorescence, colour changes, or Fabry–Pérot techniques) which are commonly used in food preparation and medical measurements. These devices, however, cost thousands of pounds. A low-cost approach, albeit a less convenient one, is simply to measure the initial sample temperature, heat the sample for a period, then remove it from the oven and re-introduce the temperature sensor.

### 4. Calibration and data reduction

Since the radiation field within a given oven will not be known *a priori*, especially if the sample is placed within an absorbing structure, the field in which the sample is to be immersed needs to be measured. Materials (see table 1) with known dielectric and thermal properties were placed in the oven and the temperature rises recorded. The same location within the oven cavity was used each time (since fields can vary substantially, even with turntables or mode stirrers).

For large samples around room temperature, a commercial pocket digital thermometer (Mannix HDT303K, with a relative inprecision of  $\pm 0.1$  K) was used to determine the temperature increase, by measurements before and after. Additionally, a digital voltmeter was used to measure the potential difference across a 1N4148 silicon diode (the voltage across this conveniently small and inexpensive device is proportional to the temperature). This diode thermometer was calibrated against known laboratory thermometers at various temperatures (in any case, this technique relies only on differential measurements, so absolute calibration is not essential) and was accurate to within  $\pm 0.5$  K. Different diodes, even from the same batch, gave slightly different voltages for a given temperature and so required separate calibration.

The small size of the diode makes it equilibrate with its surroundings rather more quickly than do other thermometers to hand and it is able to operate at far lower temperatures than can typical pocket thermometers. That said, other (more expensive) laboratory temperature sensors such as platinum resistance thermometers or thermocouples would work equally well.

The temperature rise relates to the absorbed power  $P$  thus:

$$P = V c_p \rho \frac{dT}{dt} \quad (1)$$

with  $V$  the sample volume,  $c_p$  the sample heat capacity and  $\rho$  the density. For most materials (see table 1) the  $c_p \rho$  product is around  $2 \times 10^6 \text{ J m}^{-3} \text{ K}^{-1}$  (e.g. kerosene with  $\rho = 800 \text{ kg m}^{-3}$  and  $c_p = 2150 \text{ J kg}^{-1} \text{ K}^{-1}$ ), notable exceptions including water-rich and porous materials. If the material has unknown thermal properties, the  $V c_p \rho$  product can be determined by heating with a small electrical resistance heater of known power.

For a plane-parallel sample geometry (an adequate approximation for these measurements and one that is easy to visualize), with normally incident power density  $I$ , area  $A$ ,

**Table 1.** Calibration materials.

Sample	Material	Density (kg m <sup>-3</sup> )	Heat capacity (J kg <sup>-1</sup> K <sup>-1</sup> )	Dielectric constant	Loss tangent	Reference
1	Teflon	1700	2200	2.1	0.0001–0.0002	[1]
2	Vaseline	800	2200	2.16	0.0004–0.0008	[1]
3	Plexiglass	1190	1700	2.6	0.003–0.008	[1]
4	Kerosene	800	2150	2.2	0.003–0.006	[1]
5	Balsa wood	70	2800	1.22	0.006–0.011	[1, 12]
6	Nylon	1150	1700	3.0	0.01–0.03	[2]
7	Corn oil	910	2000	2.5	0.03–0.1	[2, 12]
8	Ivory soap	900	1800	2.97	0.12–0.34	[1]
9	Pure water	1000	4200	77	0.15	[2]
10	1% NaCl solution	1010	4200	77	0.3	[2]
11	5% NaCl solution	1050	4200	67	0.8	[2]

sample thickness  $t$  and absorptivity  $\alpha$  the power  $P$  absorbed by the sample is as follows:

$$P = IAF(1 - e^{-\alpha t}) \quad (2)$$

where  $F$  is a correction factor to account for reflection at the sample edge: the value may be in general assumed to be between unity and the Fresnel transmission coefficient  $[(\epsilon^{0.5} - 1)/(\epsilon^{0.5} + 1)]^2$  where  $\epsilon$  is the real part of the complex dielectric constant. In practice, the sample container provides some anti-reflection impedance matching between free space and the sample, so more than the Fresnel coefficient is permitted into the sample. Additionally, multiple reflections within the sample increase the average path length beyond  $t$ . For all except high- $\epsilon$  materials (generally, water-rich ones) a value of unity may be safely assumed for  $F$ .

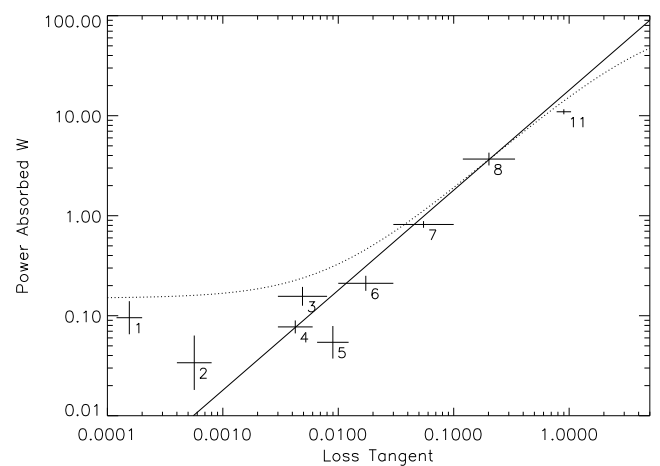
The loss tangent  $\delta$  is defined as  $\tan \delta = \epsilon''/\epsilon'$ , where  $\epsilon''$  is the imaginary part of the complex dielectric constant (also termed the loss factor in the food literature [2]). It relates to the absorptivity thus:  $\alpha = 2\pi\epsilon''/(\lambda\epsilon^{0.5})$ , with  $\lambda$  the free-space wavelength (0.12 m for domestic microwave ovens). Provided that the sample optical depth  $\alpha t$  is small ( $<1$ , to within about 50% accuracy) expression (2) may be considered linear,  $\alpha t \simeq (1 - e^{-\alpha t})$  and hence the power absorbed by the sample is proportional to the loss tangent. For the accuracies under consideration here, the  $\epsilon^{0.5}$  term in the absorptivity definition may be ignored too and the parameters  $\epsilon''$ ,  $\alpha$  and  $\delta$  may all be considered proportional to  $P$ .

Results are shown in figure 1. In general the absorbed power and the loss tangent line up quite well and, for the oven (a roughly 600 W device) and sample geometry (3 cm diameter, 5 cm long cylinders) used, the constant of proportionality ( $P/\delta$ )  $\simeq 20$  W.

## 5. Application to planetary ices

The relative transparency of low-temperature ice to radio and microwave radiation is well known. This radar transparency can give rise to unusual radar echoes [6] from the icy satellites of Jupiter, the Martian poles and certain ice deposits in Greenland [7]. These surfaces exhibit surprisingly high reflectivities and unusual polarization ratios, indicating that high-order volume scattering, facilitated by the transparency of the material, is responsible.

The most remote object detected by radar [8] is Saturn's satellite Titan, to be studied by the NASA-ESA Cassini



**Figure 1.** The measured heat absorption (crosses; numerals refer to table 1) correlates with loss tangent, although errors become large for transparent materials ( $\delta < 0.005$ ). Horizontal lines forming the crosses indicate ranges of literature values for  $\delta$ ; vertical lines indicate uncertainty in absorbed power due to uncertainty in  $c_p\rho$  and temperature-difference-measurement errors. The full line indicates an ideal correspondence; the dotted line indicates schematically the limitations of the method for a given sample size due to heat leakage for low  $\delta$  and 'saturation' at high  $\delta$ .

mission. Although Titan's optical reflectivity resembles that of ice-rich satellites like Ganymede, its radar reflectivity is rather lower. As discussed elsewhere [9], this may be due to some combination of rock, ice and organic components. Another possibility is that the ice forming Titan's surface may be ammonia-rich. Ammonia-rich ice has a higher real dielectric constant [9] than does pure water ice, so it transmits less. It has also been indicated qualitatively that ammonia-rich ice is more absorbing than pure ice at  $\simeq 1$  MHz frequency. It may be expected that ammonia-doped ice is extremely absorbing at gigahertz frequencies, due to the strong absorption by the inversion of the ammonia molecule. The methodology outlined above is here applied to test this hypothesis.

Ice samples were prepared by freezing distilled water and water spiked with 0.2% and 1% ammonia (cryovolcanic flows on Titan may include up to 30% ammonia [9]) in 200 ml Tupperware containers. These containers have walls of polypropylene, which is weakly absorbing. The samples were placed in a cooler containing dry ice overnight to freeze them and attain a steady temperature of  $-80^\circ\text{C}$ . The larger containers were used to try to minimize heat-leakage effects: these containers were calibrated using materials 4

and 7 for convenience. After the samples had been made, 3 mm diameter holes were drilled about 3 cm deep to allow temperature measurements at several places.

The temperature of the sample was determined by placing diodes into the holes. It took around 1 min for the diode to equilibrate with its surroundings. An important consideration in these measurements (and, indeed, in the preparation of food) is that ice exhibits a strong increase in absorptivity with temperature, due to the increase in water content and the much higher absorptivity of water ( $\delta \simeq 0.2$ , compared with  $<0.003$  for ice). Thus, if a small part of the sample were to be made warmer than the rest, by introducing a warm thermometer, for example, then it will preferentially absorb microwave energy and warm up, accelerating the effect, termed 'thermal runaway'. Thus the diode thermometers were pre-chilled in dry ice before introducing them into the sample.

The samples were heated in the oven in the same way as the calibration materials: around 3 min was appropriate for pure ice, whereas the ammonia-rich samples tended to melt partially if heated for this duration, so typically 30–60 s was used. The temperatures were measured with the diodes afterwards.

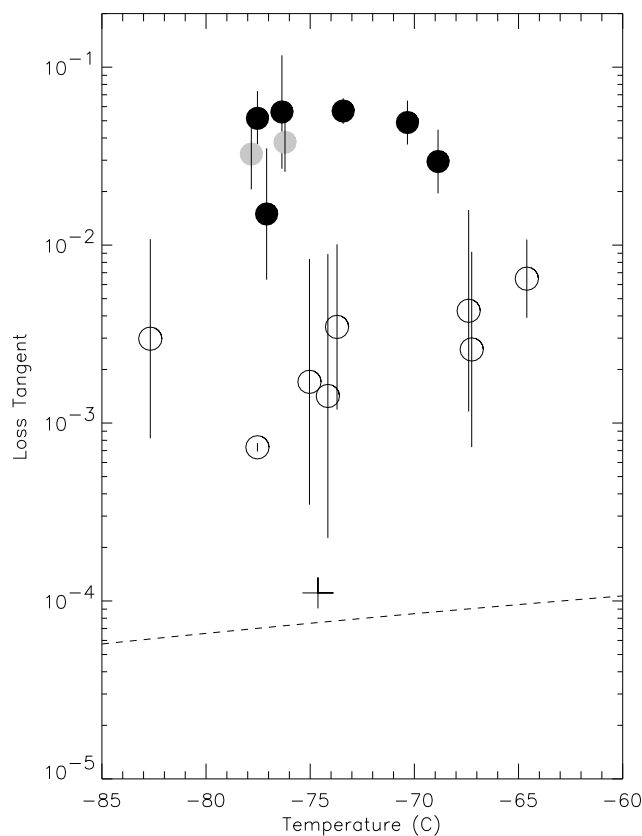
It may be safely assumed that, within the accuracy of the experiment, the density and heat capacity of the ice were not appreciably changed by the presence of traces of ammonia. Thus the considerable enhancement of heating corresponds to a similar enhancement of the absorptivity compared with that of pure ice.

The results are shown in figure 2. It can be seen that the absorptivity of the ammonia ice is 1–2 orders of magnitude higher than those for ice. The ice measurements are in turn about one order of magnitude higher than models [10] predict and then values measured by other techniques [11]. The fact that pure ice is below the measurement floor for this particular experiment does not affect the ammonia measurements: the inferred loss tangents for this material may be confidently used in radiative-transfer calculations.

Possible enhancements to the technique to improve accuracy are to chill the oven and insulation material, such that heat leak into the sample is eliminated. Control experiments with lower or zero oven power could help quantify this error source. Additionally, the 'floor' to the measurement technique may be in part the microwave energy absorbed by the container walls. Using a thinner-walled container and/or one made of a particularly microwave-transparent material would ameliorate this error source

## 6. Conclusions

I have demonstrated that the temperature rise in a material irradiated in a microwave oven can be related to its microwave absorptivity and that useful constraints on this quantity can be obtained with very rudimentary equipment. This method may be useful for rapid investigations and to identify materials that merit more detailed investigation by conventional and more accurate techniques, for field investigations (most convenience stores and gas stations are equipped with microwaves sources), for educational projects (since the method requires an understanding of the



**Figure 2.** Inferred loss tangents for pure ice (open circles) 0.2% (grey) and 1% (black) ammoniated ices. The broken line is a model [10] for pure ice. The cross is a conventional measurement [11] (at 1.25 cm wavelength). The pure-ice measurements are clearly too high and indicate a limiting sensitivity of the method, but the high absorption of the ammonia samples is clear.

propagation of electromagnetic radiation in materials) and for situations in which materials are awkward to handle. The technique has been used to show that cold water ice spiked with ammonia is far more absorbing than is pure ice.

## Acknowledgments

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