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The electrodeless discharge lamp: a prospective tool for photochemistry
Part 2. Scope and limitation

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Abstract

We have previously reported results from the study of an original photochemical reactor consisting of an electrodeless discharge lamp (MWL) placed into the reactor vessel of a microwave oven [P. Klán, J. Literák, M. Hájek, J. Photochem. Photobiol. A: Chemistry 128 (1999) 145]. The microwave field generates ultraviolet irradiation in the lamp at the same time as it interacts with the studied sample, which is therefore affected by a simultaneous UV/VIS and MW irradiation. Here, five different common photoreactions have been investigated in the reactor: Norrish type II reaction of valerophenone and its *p*-methyl derivative, photo-Fries reaction, photoreduction of acetophenone, photolysis of a phenacyl ester, and a radical nucleophilic substitution of chlorobenzene in methanol. The reaction conversions and product distributions have been studied in terms of the MWL quality and the scale of the experiment. In addition, MW experiments were compared to those using a conventional UV lamp. The electrodeless lamp is presented as a very simple, economic, and efficient tool for photochemistry.

Keywords: Microwave photochemistry; Electrodeless discharge lamp; Photo-Fries reaction; Type II reaction; Photoreduction

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1. Introduction

It has been known for several decades that the electrodeless discharge lamp (microwave lamp; MWL) generates ultraviolet (UV) radiation in the microwave (MW) field, i.e. by wireless way [2,3]. Církva and Hájek [4,5] have proposed a simple MW-UV reactor for potential applications in organic chemistry by placing MWL into the reaction vessel in a modified microwave oven. We recently reported results from the first systematic investigations in this reactor [1]. The technique provides a very simple, economic, and efficient design that results in a simultaneous MW and UV irradiation of the reagents, undergoing photochemical reactions under extreme thermal conditions. Other examples of the MW effect on photochemical reactions are rare. Chemat et al. [6] studied the rearrangement of *o*-aryloxyacetophenone and the degradation of humic acid in an original microwave-ultraviolet combined reactor. It was also suggested that microwave-assisted photodegradation of pollutants may be of principal interest in the future [7].

Energy of MW radiation ($\approx 1\,\mathrm{J\,mol^{-1}}$ at 2.45 GHz) is more than five orders of magnitude lower than that of UV radiation ($300–480\,\mathrm{kJ\,mol^{-1}}$), and therefore not sufficient to disrupt bonds of common organic molecules. Chemical processes performed under microwave radiation (microwave chemistry) are believed to be affected by superheating, polarization, dielectric properties, hot spots formation, nuclear spin rotation, and spin alignment [8–14]. However, the existence of a ‘specific microwave effect’ in homogeneous reactions is still a matter of debate. We hope that microwave photochemistry may bring a deeper understanding to the topic.

In this paper, microwave-assisted photochemistry of several common photoreactions is described in terms of MWL quality and scale of the experiment. In addition, MW photochemical experiments are compared to those using a conventional UV lamp.

2. Experimental section

2.1. Equipment

^{1}H and ^{13}C NMR spectra were obtained for solutions in $CDCl_3$ on an Avance Bruker DRX 500. Gas chromatography was achieved on a Shimadzu GC-17A apparatus, a GC/mass system TRIO 1000 (Fisons Instruments), and on an HP 6890 gas chromatograph.

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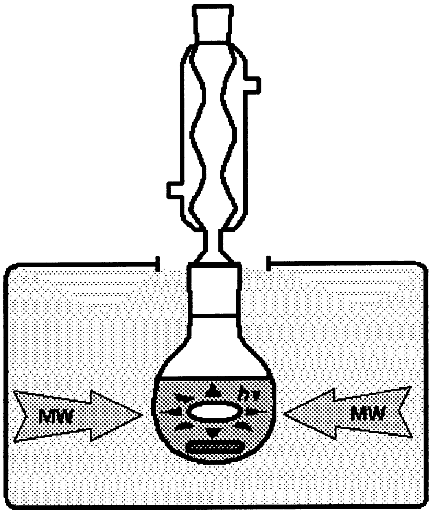

Fig. 1. A simplified scheme of an MW reactor consisting of MWL and the stir bar placed into the reaction mixture in an MW field.

MW experiments (Fig. 1) were accomplished in the modified MW oven (Samsung M746, 800 W[1]) with holes drilled to the oven walls: one hole for a condenser tube; and an inlet and outlet port for a tube with circulating water. A part of the oven bottom was replaced by an aluminum plate, carefully attached to the framework, to allow magnetic stirring. Since the microwave equipment must operate within the safety limits prescribed locally in the country of installation [16], the limit on the safe stray leakage of microwave power density was kept below 10 mW cm^{-2} at 2450 MHz measured at the 50-mm distance from the equipment.[2] *It was extremely important that the equipment was checked for leaks, especially around the modified area.* Some studies were also conducted in a microwave instrument Synthewave 402 (Prolabo) equipped with an IR pyrometer (not calibrated), quartz reaction vessel, and a cooling system. This microwave reactor operated with maximum of 300 W output power at 2450 MHz frequency.

Electrodeless discharge mercury lamps (MWL) were manufactured in Teslamp (Prague, the Czech Republic). The lamps were made of 9–14 mm quartz or Simax (Pyrex equivalent) tubing (of ≈1-mm thick glass) of 13–37-mm length, filled with mercury and argon, and sealed under 20 Torr vacuum. Simax absorbs most of UV irradiation below 280 nm.

2.2. Chemicals and solutions

Valerophenone (>99%) was obtained from Aldrich Chemical Co. and was further purified by distillation under reduced pressure. *p*-Methylvalerophenone was synthesized from *p*-methylbenzonitrile and butylmagnesium bromide according to the Wagner's procedure [17,18] and was purified by distillation under reduced pressure. Acetophenone, *i*-propanol, and chlorobenzene (Lachema Co.) were purified by distillation. Phenyl acetate (>99%) was purchased from Aldrich and was used as received. Phenacyl ester of benzoic acid was synthesized from α-bromoacetophenone according to the literature and purified by recrystallization [19]. Hydroxyacetophenones (>98%) were purchased from Aldrich and served as analytical standards as received. Hexadecane (>99%) from Schuchardt was used as received. All solvents were purified by standard procedures.

2.3. Microwave and irradiation experiments

The experimental apparatus consisted of an electrodeless discharge lamp (MWL) placed into the reaction solution in the MW field. We assured that most of the solution was under direct MW radiation. In the Synthewave chamber, the vessel equipped with a condenser rotated under argon atmosphere. The MW power and temperature was controlled by Prolabo software.

A typical arrangement (Fig. 1) in the modified oven involved a reaction vessel (50 ml) with a reaction mixture (20 ml), equipped with a Teflon-covered stir bar and MWL (larger lamps usually float in liquids while smaller ones sink). This allows irradiating, the whole volume of the solution by UV light. The vessel was connected to a highly efficient water-cooled condenser by a 100–300 mm long glass tube. The microwave power was adjusted to a maximal value (800 W), which guaranteed a continual MW radiation. Circulating cool water was used in cases when a small amount ($<$50 ml) of a polar liquid or any amount of a non-absorbing (non-polar) liquid was used. It removed the excess of microwave power and prevented the magnetron from destruction by overheating. The volume of liquid remained the same at the end of every experiment as it was in the beginning. All experiments were kept under argon atmosphere.

Working with superheated flammable and toxic solvents in a microwave reactor needs a special attention.

The comparative conventional irradiation experiments were carried out in either quartz or Simax 13 mm × 100 mm tubes, equipped with a condenser, placed at a distance of 60 mm from a conventional medium-pressure mercury lamp (125 W, Teslamp). The 5 ml solutions were degassed by bubbling the solutions with argon for 15 min before irradiation.

2.4. Photoproduct identification and analysis

The only photoproducts followed for all reactants in Norrish type II studies were acetophenones and cyclobutanols. Formation of a minor photoproduct ($<$2% yield, assuming the same GC response factor as valerophenone) was also observed and it was believed to be *1*-arylcyclopentanol as it was described elsewhere [18,20–26]. Identification of acetophenones and starting valerophenones, isolated by flash chromatography, was based on ^{1}H NMR, ^{13}C NMR, and

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[1] The effective power of the oven in the position of the reaction vessel was experimentally found to be ≈300 W according to Watkins [15].

[2] The oven was modified by Radan Co., Živanice, Czech Republic.

on GC comparisons with authentic samples (Aldrich). Cyclobutanols were identified using GC and GC/mass instruments [20–26]. The same analysis was accomplished for *p*-methylvalerophenone. Concentrations of the photoproducts were calculated from peak integrations of data obtained with flame ionization detection, assuming that the cyclobutanols had the same response as valerophenones. We tested the stability of cyclobutanols in boiling solvents. The reaction mixtures, after irradiation at 20°C, were heated to 80°C for 4 h (eight times as long as an average photochemical experiment) and re-analyzed. Concentrations of the photoproducts remained unchanged in all cases.

Photo-Fries [27,28], phenacyl ester photolysis [20] and photoreduction [30] reaction photoproducts were analyzed by GC/MS and GC methods. Identification of the products was based on comparison of their mass spectra and retention times with those obtained from authentic samples. In photoreduction reactions, only acetophenone disappearance was followed.

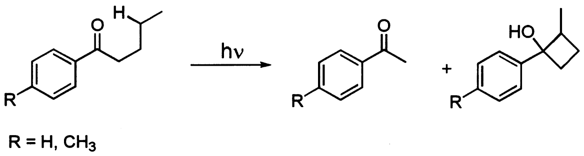
Photochemistry of chlorobenzene in methanol was analyzed by GC/MS with hexadecane as an internal standard [31]. Identification of photoproducts was carried out by GC/MS chromatograph, equipped with the mass selective detector HP 5972 (Hewlett-Packard, USA). The mass spectrometer operated in the scan mode using EI ionisation; the mass spectra were collected in the mass range 50–550 *m*/*z*.

Each sample was analyzed three times; in case that the values differed by >8% the measurement was repeated. In all MW-UV experiments, the photoproduct distributions were found to be almost identical with those obtained at 20°C.

3. Results and discussion

3.1. The electrodeless discharge lamp: use and limitation

Hájek et al. [5] reported that the reaction conversion under an MW-UV condition relates to the size of the electrodeless lamp. For this paper, we have accomplished an investigation of the Norrish type II reaction [20–25] in terms of (1) the number of MWLs applied, (2) the MWL size, and (3) the volume of a reaction mixture. Valerophenone derivatives with hydrogen on γ-carbon react on their alkyl chains via the triplet state to produce 1,4-biradicals, which can cleave, cyclize (Scheme 1), or disproportionate back to the starting ketone. [20–25] The only photoproduct, acetophenone, was followed by GC since the product distribution was found to be practically unaffected by MW conditions [1].



Scheme 1.

Table 1
The use of MWL

MWL	VP conversion[b] (%)	MeVP conversion[b] (%)	Heating rate (K s^{-1})
Number			
0[a]	–	–	<0.001
1[a]	5.84	5.23	0.057
2[a]	9.62	8.55	0.126
3[a]	17.66	16.31	0.216
Size			
Small[c]	0.78	–	0.057
Large[c]	13.69	–	0.827

[a] The number of quartz MWLs (size (volume) = 1.67 cm^3). Synthewave 402 operated at 80 W output power.

[b] Conversion of the type II reaction in cyclohexane measured as acetophenone or *p*-methylacetophenone formation, respectively.

[c] Simax MWL: the *small* lamp was of 1.17 cm^3 and the *large* lamp of 5.05 cm^3 volume. Samsung M746 operated at 800 W output power.

Table 1 lists results from comparative studies of type II photochemistry in a MW reactor. A series of experiments with 1, 2, or 3 identical MWLs, respectively, placed in one reaction vessel have been carried out. Photochemistry of an equimolar mixture of valerophenone (VP) and *p*-methylvalerophenone (MeVP) in cyclohexane showed a linear (additive) increase in the reaction conversion with every additional lamp. For comparison, the rate of heating (heating was caused by the lamp operation only because cyclohexane practically does not absorb MW radiation [1]) was monitored (Table 1). Although the lamp gives over three times as much UV radiation as the conventional electrode lamp, infrared loss is still large [2,3]. The rate was calculated from a linear 'temperature versus time' dependence in the 25–50°C interval. Temperature was detected by an IR pyrometer and a proper operation of the lamp was observed through the instrument window. As in the type II experiments, nearly the same additive effect on the rate for each lamp was found: the increase in conversion was perfectly proportional to the increase in heating rate. Thus, a monitoring of the IR output may be useful, especially when a precise control of the reaction is of interest (i.e. without the necessity to discontinue irradiation).

Two MW lamps of a different size were tested for the type II conversions as well as for the rate of solvent heating. Table 1 shows that the larger lamp was a much more effective source of UV radiation than was expected from its size. The conversion increased approximately by a factor of 18, but the volume only by a factor of 5. Thus, larger lamps are *more economic* sources of radiation.

In search for limitation of the MW-UV experiments, we carried out photochemistry in different volumes of the reaction mixture. The valerophenone type II reaction was chosen again to ascertain the experimental consistency. Fig. 2 shows a typical linear '*volume versus photoproduct amount*' dependence. However, the dependence for larger volumes leveled off with an increasing volume within the Beer–Lambert law.

We demonstrated in our previous paper that polar solvents absorb most of the MW radiation; therefore, the lamp

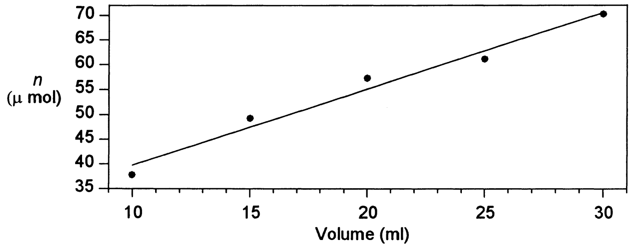


Fig. 2. Dependence of the amount of acetophenone (*n*) formed by the photolysis of valerophenone (0.1 M) in cyclohexane on the reaction mixture volume. Conversions were kept below 4% to avoid the photoproduct interference.

operation is not very efficient [1]. In addition, our experiments were troublesome in Synthewave 402 because the cooling system was not designed for volatile liquids. A modified MW oven was found to be much more useful. Such a commercially available oven equipped with an efficient condenser and modified to enable magnetic stirring or to use large-volume reaction vessels has been already adopted for microwave chemistry [8–12].

We tried to compare MW-UV experiments carried out in different polar solvents (acetonitrile, ethanol, and water). Acetonitrile ($\mu = 3.44$ D) was found to be still a suitable solvent. The lamp never ceased to operate and the photochemical conversions were adequate. With increasing MW output power, the solvent absorption ability was saturated and the remaining energy served for the lamp operation. For experiments in ethanol ($\mu = 1.66$ D), it was sometimes necessary to place MWL at least partially above the liquid surface to guarantee a proper lamp functioning. *MWL has to be always fixed in the position in which the reflux efficiently cools it down. The lamp overheating causes the lamp emission failure.* The comparison of photochemistry when the lamp was placed either into or above the reaction mixture is shown in Table 2. It is clear that only a small portion of light coming from the lamp placed outside the solution is absorbed by the reagents. Water ($\mu = 1.84$ D) was found to be a rather impractical medium. The lamp did not fully operate inside as well as above the liquid. Here, an external source of UV irradiation for such MW-UV experiment is recommended.

Two different MW lamps, made from either quartz or Simax tubing, have been tested in order to determine filter capabilities of the material (thanks to 1-mm thick walls, the wavelength of UV radiation coming from Simax MWLs was considered to be >280 nm). The spectral output of the electrodeless discharge mercury lamp is known to resemble more those of high-pressure mercury lamps [32]. Thus, the conversion using a Simax lamp was lowered approximately by a factor of 7 compared to that of a quartz lamp (Table 3), which is in a good accord with well-known NIST data of irradiance from the Hg(Ar) lines [33]. When a high-energy band at 253.65 nm was filtered off, the radiant power coming from the lamp was significantly lowered. Since the molar absorption coefficients of valerophenone at 254 and 280 nm are nearly the same and since the product distribution was essentially unchanged whatever glass material was used, we assume that the conversions correspond to the filter effect only. In addition, the nucleophilic photosubstitution of chlorobenzene in methanol [34] (Scheme 2a) was carried out with those two MW lamps. Practically no reaction was observed when the Simax lamp was used (chlorobenzene significantly absorbs below 280 nm); however, the quartz lamp provided anisole production. *The results suggest that the choice of the material for the lamp is crucial and can be tailored according to photochemical needs.*

Table 2
An example of the type II photochemistry in ethanol

MWL[a]	VP conversion (%)[b]
Placed into the solution	21.6
Placed above the solution[c]	0.06

[a] Samsung M746.
[b] Conversion of the reaction was measured as acetophenone formation.
[c] Approximately 100 mm above the liquid surface.

Table 3
Quartz vs. Simax MWLs of the same size

MWL	MW output power[a]	VP conversion (%)[b]
Quartz	15	8.18
Quartz	50	20.32
Quartz	80	28.74
Simax	15	0.47
Simax	50	1.52
Simax	80	2.06

[a] Synthewave 402.
[b] Conversion of the reaction was measured as acetophenone formation.

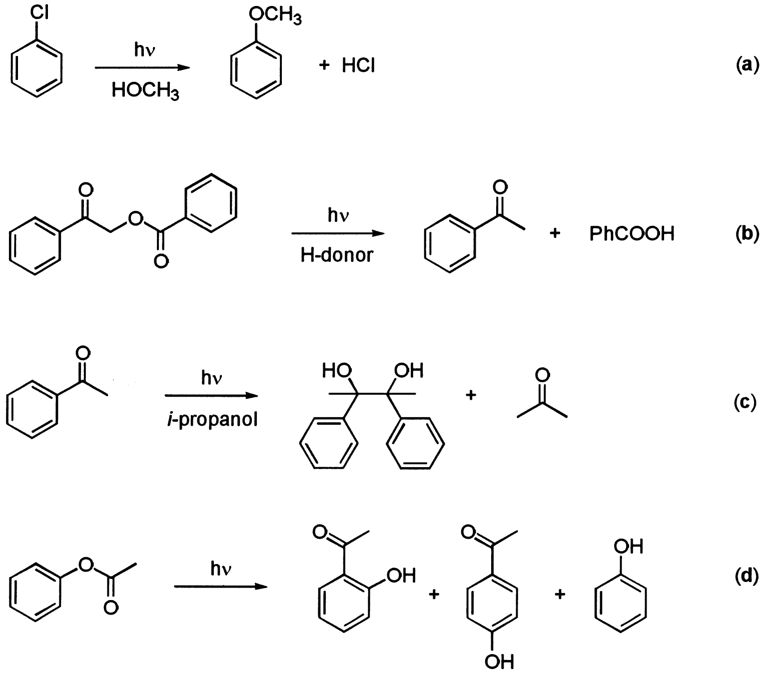
3.2. Comparison of the MWL experiment to the conventional irradiation

Several photochemical reactions, using both MWL as well as a 125 W conventional medium-pressure mercury lamp, have been studied. We have not attempted to measure absolute quantum yields in the MW-UV experiments, because of experimental difficulties. It is known that some photochemical reactions are sensitive to temperature. That is why parallel experiments in boiling solvents were carried out to determine possible temperature effects on the reactions.

Five different reactions were studied in terms of the relative photochemical efficiency and the product distribution:

1. the type II reaction of valerophenone (Scheme 1);
2. photolysis of phenacyl benzoate [29] (Scheme 2b);
3. photosubstitution reaction of chlorobenzene in methanol (Scheme 2a);
4. photoreduction of acetophenone by *i*-propanol [30] (Scheme 2c); and
5. photo-Fries reaction of phenyl acetate [27] (Scheme 2d).

Low concentrations of reactants reduced the probability of intramolecular reactions as well as intermolecular energy



Scheme 2.

transfer. Intramolecular triplet energy transfer in phenacyl benzoate is expected to be endothermic and, thus, negligible [35,36].

The principal aim of the comparative studies was to receive an impression of MWL irradiation capabilities for photochemistry. Table 4 presents results of various experiments using either quartz or Simax lamps of different sizes. A large quartz MWL seemed to be somewhat more efficient than a Simax lamp of the same size when compared to an external (conventional) source of radiation of the same wavelength (items 2, 3, 5 vs. 4, 6). The table suggests that photochemical efficiency of the mentioned reactions, using the conventional 125 W mercury lamp under our experimental conditions, was approximately of the same magnitude as in experiments in which a *large* MWL was applied. It is worth emphasizing that the conversion of most presented

Table 4
Microwave photochemistry vs. conventional irradiation[a]

	Reaction mixture (wavelength used)	Reaction conversions (%)	
		Conventional 125 W lamp[b]	MWL (size)
1	The type II reaction of 0.1 M valerophenone (Scheme 1) in cyclohexane (>254 nm)	19.1[c]	6.8 (*small*)[c]
2	Photolysis of 0.01 M phenacyl benzoate (Scheme 2b) in the presence of Et_3N in benzene (>280 nm)	11.6 (18.3)[d]	8.7 (*large*)[d]
3	Photolysis of 0.01 M phenacyl benzoate (Scheme 2b)in the presence of benzhydrol in benzene (>280 nm)	3.0 (4.3)[d]	4.5 (*large*)[d]
4	Photosubstitution of 0.005 M chlorobenzene (Scheme 2a) in methanol (>254 nm)	48.7 (8.7)[e]	91.0 (*large*)[e]
5	Photoreduction of 0.1 M acetophenone by *i*-propanol(Scheme 2c) in cyclohexane (>280 nm)	84.9 (59.4)[f]	38.0 (*large*)[f]
		16.2[g]	33.5[g]
6	Photo-Fries reaction of 0.01 M phenyl acetate (Scheme 2d) in methanol (>254 nm)	4.2[h]	8.1[h] (*large*)
		12.0[i]	25.4[i]

[a] All MW-UV experiments were carried out in Samsung M746 at 800 W output power. The reproducibility of the measurements was ±8; however, the MW experiments were sensitive on the amount of circulating water in the oven removing the excess of MW radiation. Thus, we estimate that the actual reproducibility in the comparative studies is somewhat higher.

[b] Irradiated for the same time period as the MW-UV experiments. Experiments were carried out in the *boiling* mixtures (the reaction vessel was placed in the 60 mm distance from the conventional lamp). The values in the parentheses were obtained in irradiation experiments at 20°C.

[c] The reaction conversions were measured as acetophenone formation.

[d] The reaction conversions were measured as acetophenone formation.

[e] The reaction conversions were measured as anisole formation.

[f] The reaction conversions were measured as acetophenone photodegradation.

[g] The photo-Fries reaction was followed as phenyl acetate degradation.

[h] The photo-Fries reaction was followed as phenol formation.

[i] The photo-Fries reaction was followed as hydroxyacetophenone (the sum of *o*- and *p*- isomers) formation.

Table 5
Advantages and disadvantages of the electrodeless discharge lamp use in photochemistry

Advantages	
1	Simultaneous UV and MW irradiation of the sample
2	Possibility to carry out photochemistry at high temperature
3	Good photochemical efficiencies
4	Simplicity (‘one-pot’ arrangement) of the experiment
5	A low cost of the electrodeless discharge lamp
6	Use of a commercially available microwave oven
7	The sample absorbs maximum of UV light when MWL is placed into the reaction vessel
8	A ‘wireless’ MWL operation
9	The choice of the MWL material may modify its spectral output
Disadvantages	
1	Impossibility to carry out photochemistry at temperatures below the solvent boiling point
2	Necessity of a special attention when working with flammable and toxic material in the MW oven
3	MWL overheating causes the lamp emission failure
4	Polar solvents absorb most of the MW radiation

reactions was temperature-dependent. However, the photoproduct distribution was found to be comparable under all experimental conditions.

The aim of this paper was to evaluate whether the electrodeless discharge lamp could be useful for photochemistry. Our study has displayed that the lamp can be a useful and readily available tool and, what is more, it can be used in studies, in which simultaneous UV and MW irradiation of the sample is needed. Since similar photoproduct distributions were observed in all different photoreactions, MW-UV conditions could be beneficial in synthetic organic photochemistry. Thanks to its reasonable photochemical efficiency, MWL may be part of a reactor designed for photodegradation of environmentally unwanted compounds. The general conclusions, based on results from this and the previous [1] papers, are listed in Table 5.

Our further research will be focused on detailed study of the microwave-assisted photoreactions in terms of quantum yield measurements and a search for possible non-thermal effects of microwave radiation.

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