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A Light Scattering and Electron Microscope Examination of Monodispersed Metal Iodate Hydrosols*

M. J. Herak, J. Kratohvil, M. M. Herak and M. Wrischer

Institute »Ruđer Bošković«; Department of Physical Biochemistry, »Andrija Štampar« School of Public Health, Faculty of Medicine; Laboratory of Physical Chemistry, Faculty of Science, University of Zagreb, Zagreb, Croatia, Yugoslavia

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Monodispersed lanthanum and lead iodate hydrosols were prepared and their properties examined by means of light scattering, electron and light microscopy, and measurements of rate of deposition of particles. The sols exhibited brilliant colour bands of higher order Tyndall spectra indicating the presence of spherical particles very uniform in sizes. Lead iodate sols, otherwise very unstable, could be stabilized by simple filtration through filter paper. This was accompanied by reversal of charge carried by the particles. The growth of lanthanum iodate was much slower. Angular distribution of light scattered by metal iodates showed typical features of monodispersed systems with spherical particles several hundred millimicrons in radius. This was confirmed by electron microscopy. Particles of a typical lanthanum iodate sol were about 700 m μ in radius, and those of lead iodate about 860 m μ . The variations in particle sizes from different preparations were considerable, and could be explained by the uncontrolled influence of the direct mixing of reacting solutions. A tentative mechanism of the formation of monodispersed metal iodate hydrosols was proposed.

INTRODUCTION

Over the last fifteen years a good deal of work on uniformly dispersed systems has been done, and much evidence has accumulated so that it became possible to formulate a theory and mechanism of the formation of such systems.^{1, 2} The experimental research in this field has been concerned primarily with the formation of monodispersed aerosols³ and sulphur hydrosols,⁴⁻¹² and with polystyrene and polyvinyltoluene latexes¹³⁻¹⁷ with very uniform particle sizes. These dispersions were mostly characterized by means of light absorption and light scattering measurements. It should be emphasized that the investigation of light scattering in monodispersed colloids has contributed decisively to final proof of the validity of the Mie theory¹⁸ of light scattering by spherical particles of any size and any index of refraction.

However, little has been done in this field of research using the inorganic colloids (e. g., slightly soluble metal salts) and their monodispersed preparations as the object of investigation. In the relevant literature we could find a very limited number of papers, except those of LaMer on sulphur sols, dealing with the inorganic colloidal dispersions of uniform particle size.

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Andreasen¹⁹ described the preparation of monodispersed suspensions and sols of barium sulphate, copper oxide, mercuric iodide and thallium iodide in particle size range from 0.14 to 4 μ . The precipitation was performed by direct mixing of the reacting components and also from homogeneous solutions. The dispersions were characterized by means of microphotography and, in one case, with the electron microscopy. No optical measurements were performed. Ginell et al.²⁰ have observed that in dilute ethanol solutions containing silver and chloride ions monodispersed silver chloride sols were formed showing several bands of higher order Tyndall spectra characteristic for spherical particles of uniform size. Unfortunately, except visual observations, they did not perform any quantitative optical and similar measurements. Interesting results were described by Berestneva and Kargin.²¹ By means of electron microscopy they characterized a sol of As_2S_3 containing spherical particles of uniform size of about 0.6 μ in diameter.

Working on a study of the precipitation of some slightly soluble metal iodates in aqueous media²² we have noticed the appearance of typical higher order Tyndall spectra. Since this indicated the presence of spherical particles of very uniform size, we have decided to investigate such systems by means of most suitable methods, i. e., the electron microscopy and light scattering. A preliminary communication on the preparation and some optical properties of monodispersed lead iodate hydrosols has been published previously.²³

EXPERIMENTAL

Preparation of hydrosols

The monodispersed hydrosols were prepared by direct mixing of a solution of potassium iodate or iodic acid with a solution of lead or lanthanum nitrate of suitable concentrations. The most convenient concentrations of the reacting solutions were found to be those on the ionic solubility sides of the precipitation curves of lead and lanthanum iodates.²² The concentrations we have used for the preparation of lead iodate were 0.001 to 0.003 N (for both components), and about 0.03 N (iodic acid) and 0.001 N (lanthanum nitrate) for the preparation of lanthanum iodate (the concentrations of all components are given for total volume of the reaction mixture).

The way of mixing of the precipitating components had great influence on the precipitation rates and on the visible optical phenomena, although Tyndall spectra could always be noticed. The optical phenomena were most distinct when one of the reacting solutions was slowly poured down the side of a glass beaker into an equal volume of the other solution. This procedure was repeated several times.

Light scattering measurements

The determination of the number and positions of the Tyndall spectra orders was performed by angular light scattering measurements using an Oster-Aminco light scattering photometer²⁴ (manufactured by American Instrument Company, Silver Spring, Maryland). This instrument was used also for the study of the kinetics of the growth of particles. All measurements were made with a cyllindrical glass cell (3 cm. in diameter) obtained from the manufacturer of the instrument. The angular distribution of the scattered light was measured in the range of angles from 35° to 135°. The desired wave lengths were isolated from a mercury spectrum (AH-4 high pressure mercury vapour lamp) by means of Corning coloured glass filters. Vertically polarized component of scattered light was measured.

Determination of the charge on particles

To obtain some insight into the character of the electric charge carried by the particles, especially in connection with the experiments on the stability of sols, a microscopic double capillary electrophoresis apparatus was used.²⁵

Electron and light microscopy

At a later stage of this work it became possible to determine the particle shape and size by means of electron microscopy. A drop of lead or lanthanum iodate hydrosols was transferred with a capillary on a film of »Mowital«. After removal of liquid, the sedimented particles were observed and photographed in a Trüb, Täuber & Co. instrument (KM-4).

Since we are dealing here with relatively large particles, it seemed convenient to prepare some photographs also with a light microscope in the usual way.

Determination of the particle size by means of the rate of deposition

This method was used previously for the determination of the particle sizes in monodispersed sulphur hydrosols, and is described in detail by Johnson and LaMer.⁶ From the velocity of settling, v, of particles on a horizontal surface from a stirred, turbulent system, the radius, r, of the particles can be calculated by means of Stokes' law:

$$r^2 = 9 \cdot \eta \cdot v / 2 \cdot g \cdot (d - d_0) \tag{1}$$

where η is the viscosity, g the acceleration due to gravity, and $d - d_0$ is the effective density of particles. The experimental technique and the treatment of results were essentially those of Johnson and LaMer.⁶ The whole procedure was tested using a monodispersed sulphur sol prepared by LaMer's prescription and grown 24 hours to a particle radius of 640 mµ. By means of the measurement of the rate of deposition we have obtained for this sol a value of 677 mµ in radius, which is in fair agreement with LaMer's value.

We could not find in the relevant literature the values of the density of lead and lanthanum iodates necessary for the calculation of the particle radius from Stokes' law. For this reason we have determined the density of lead iodate using a method described in ref. 26, pp. 264 and 288. Lead iodate was precipitated, washed, and then boiled (to assure complete wetting of the surface of particles) with several solvents (benzene, toluene, cyclohexane) in a special pycnometer equipped with a back-condenser. The mean value from ten determinations using various solvents was 6.30 ± 0.04 g. cm.⁻³ The procedure was tested on a sample of silver iodate, for which the density was reported in the literature²⁷ (5.525 g. cm.⁻³).

RESULTS AND DISCUSSION

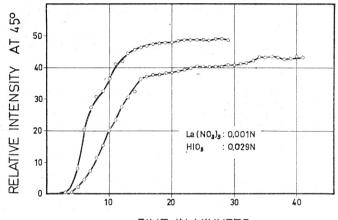
The visible optical phenomena and the growth of particles

The growth of lead iodate particles was very fast and after about a minute or two the sols exhibited equally brilliant colour (red and green) bands as LaMer's sulphur sols after 24 hours. The sols were very unstable, and after about 10—15 minutes the sedimentation took place and the colour bands began to disappear. It was therefore impossible to measure the kinetics of the growth of particles by light scattering.

The growth of lanthanum iodate particles was slower and after mixing 20—30 minutes were necessary for the Tyndall spectra to appear distinctly. An example of the kinetics of growth, as revealed through the change of intensity of light scattered at 436 mµ and at 45° relative to the direction of incident beam, is given in Fig. 1. Unfortunately, the kinetic measurements could not be evaluated more quantitatively. When measurements were repeated, the curves of the type as in Fig. 1 were obtained, but with larger or smaller differences between them. No doubt the way of mixing has a rather large influence because of random local excess concentrations unavoidable in the direct mixing of the reaction components.

Since the lead iodate hydrosols were unstable and only for a short time after mixing the Tyndall spectra could be observed, we have tried to stabilize the sols. This was particularly important for electron microscopy and angular

light scattering measurements. Gelatin in concentrations of 0.1 to $1.0^{\circ}/_{0}$ was very effective as protective colloid. Although the sol showed Tyndall spectra for many days, the colour bands were not so distinct. However, simple filtration of the sol through ordinary filter paper greatly prolonged the stability of the sol so that other measurements on it could be made. The filtration had not any influence on the intensity, number and position of the Tyndall spectra orders.



TIME IN MINUTES

Fig. 1. Time dependence of light (436 mµ) scattered at angle 45° for two monodispersed lanthanum iodate hydrosols both originated from 0.001 N lanthanum nitrate and 0.029 N iodic acid.

The filtration of the lead iodate hydrosol through filter paper was accompanied by a change of the charge of particles as determined by means of electrophoretic measurements. Before the filtration, a sol originated from the mixing of solutions of lead nitrate (0.0035 N) and potassium iodate (0.0015 N)was found to be positively charged as was expected because of an excess of lead ions in solution. After the filtration the sol particles had a negative charge. Although we have not at present a definite explanation of this phenomenon, we can draw the attention to a paper of Ultee and Hartel.²⁸ They have noted that lead ions, in comparison with other cations, exhibit the strongest affinity for carboxyl groups in filter papers. If we apply this finding to our problem we can imagine that filter paper removes the excess lead ions from solution, thus possibly preventing subsequent growth of particles originally formed and grown.

Angular distribution of light scattering

The results of angular measurements of light scattering in sols of lead and lanthanum iodate could not be evaluated quantitatively in respect to particle sizes. The great influence of mixing on the formation of precipitates frustrated any conclusive location and determination of the numbers of Tyndall spectra orders. Nevertheless, it seemed to us that an illustrative example of light scattering measurements should be included in this report. In Fig. 2 the ratio of the intensity of light scattered at two wave lengths (436 and 365 mµ) was plotted versus the angle of observation for a lanthanum iodate sols. Two wave lengths were chosen because their ratio (1.195) is very close to 1.2, thus meeting the requirements of the present tables of intensity functions available, which were constructed on the basis that $\alpha_1 / \alpha_2 = 1.2 (\alpha = 2 \pi r / \lambda)$. Similar plot is made in Fig. 3 for lead iodate using polar coordinates. For the sake of comparison, the results for a LaMer's mono-dispersed sulphur sol and for polydispersed lead iodate and sulphur sols are also included.

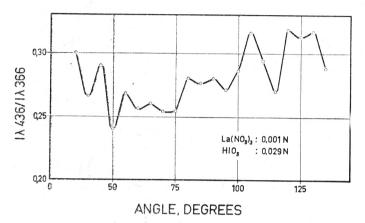


Fig. 2. Angular distribution of light scattering of the same system as in Fig. 1. Abscissae: angles in degrees; ordinates: intensity ratio at 436 and 365 mμ wave lenghts.

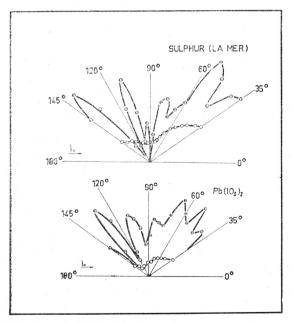


Fig. 3. Angular distribution of light scattering using polar coordinates for monodispersed (crossed circles) and polydispersed (white circles) hydrosols of sulphur and lead iodate.

The results of angular measurements of light scattering unambiguously indicate, on the basis of Mie theory of light scattering as developed by LaMer et al., that the particles of metal iodate sols investigated are spherical in shape and very uniform in size, and that the radius of spheres must be several hundred millimicrons.

The shape and size of the particles

Although light scattering measurements indicated conclusively the spherical shape of particles, it was desirable to make this indication more plausible, and also to determine the size of the spherical particles. Electron microscopy seemed as most suitable for these purposes. Fig. 4 and 5 represent characteristic examples of electron micrographs. The spherical shape and uniformity in size of the particles are evident. To test the reproducibility of the values of the size of particles, we have prepared six times the same system (lead or lanthanum iodate) in exactly the same way in regards to the concentrations and the mixing (as we could control it), and the electron microscopic pictures were taken four times for each preparation. The particle sizes varied for various

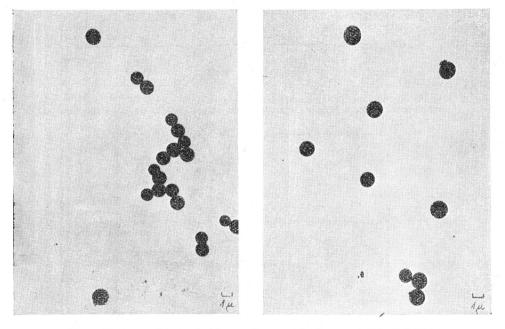


Fig. 4. Electron micrograph of monodispersed lanthanum iodate hydrosol. Fig. 5. Electron micrograph of monodispersed lead iodate hydrosol.

preparations of lanthanum iodate (prepared from 0.001 N lanthanum nitrate and 0.029 N iodic acid) in the range from 625 to $780 \text{ m}\mu$ in radius, the mean value being about $700 \text{ m}\mu$. The range of particle sizes for lead iodate preparations (prepared from 0.0035 N lead nitrate and 0.0015 N potassium iodate with subsequent filtration) was from 770 to $970 \text{ m}\mu$ in radius, the mean value being about $860 \text{ m}\mu$. As a tentative explanation for this lack of reproducibility of the preparation of metal iodate monodispersed sols, we may propose the uncontrolled influence of the direct mixing of reacting components. It should be noted that the variations for lead iodate were greater than for lanthanum iodate in accordance with much faster growth of lead iodate particles. A final answer to this question could be obtained from the study of homogeneous precipitation^{20, 30} of metal iodates. The corresponding investigations will be initiated in our laboratories in the near future.

Since the particles of metal iodates were of relatively large sizes, we have also taken some light microscopic photographs of the same systems as described above. The spherical shape and uniformity in size are evident from Figs. 6 and 7. The average radius determined from light microscopy amounted to about 730 mµ for lanthanum iodate and 810 mµ for lead iodate, in fair agreement with the electron microscopy values (700 and 860 mµ, respectively).

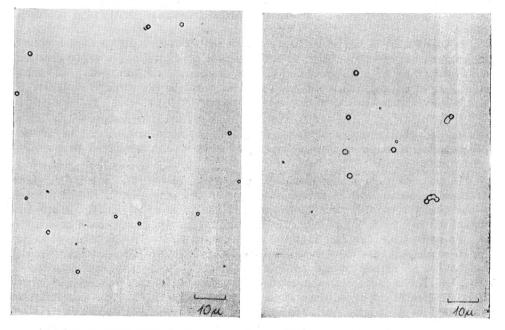


Fig. 6. Light micrograph of monodispersed lanthanum iodate hydrosol. Fig. 7. Light micrograph of monodispersed lead iodate hydrosol.

It is usual to find in electron microscopy that particles are rounded either inherently or by the action of the electron beam.³¹ Although in all electron micrographs taken we could not detect a slightest trace of the other possible shapes of the particles (only in few cases some ellipsoids of axial ratio little differing from unity were discernible), the similar process of smoothing and rounding the edges would not be improbable. However, the appearance of higher order Tyndall spectra in our systems gives a very strong evidence of the presence of spherical particles.

From the measurements of the rate of deposition of lead iodate particles by the method of Johnson and LaMer⁶ we have calculated the radii of the particles. For five separate identical preparations, the particle radius varied

from 655 to 732 mµ, the average value being 693 mµ. This value is considerably lower than that found by electron microscopy (860 mµ). Because of the relatively great velocity of settling of lead iodate particles ($v = 6 \times 10^{-4}$ cm./sec.), this method must be considered as only a rough one, which is nevertheless capable to give the approximate values for particle radii.

If we summarize the results presented above, we may accept as an experimentally established fact, that in the course of precipitation of lead and lanthanum iodates in aqueous solutions within some concentration limits the particles are formed, which are spherical in shape and so uniform in size that the appearance of beautifully coloured bands of higher order Tyndall spectra is possible. To our best knowledge, this represents a first successful attempt to obtain in a very simple way, through direct precipitation reaction between two electrolytic solutions, a monodispersed colloidal preparation.*

Two points still await for explanation, i.e. monodispersity and spherical shape.

Nowadays, it is fairly well established that whether the process involved in preparation of monodispersed colloids is a precipitation, a crystallization, a condensation, or a polymerization, the essential condition seems to be the supplying of a fixed number of nuclei which subsequently grow to much larger size with no nucleation occuring during this growth period.^{1, 2, 10}.

It is important to recognize that as small as 10 to $20^{\circ}/_{\circ}$ supersaturation is needed to produce a monodispersed colloid. Since nucleation can be accomplished at this small degree of supersaturation a monodispersed preparation will result.

If we now turn to our problem, the first question that arises is: which processes are responsible for nuclei to appear in a single burst rather than in a cascading, repetitive way, when mixing the solution of metal nitrate and potassium iodate (or iodic acid). The answer to this question should be sought, in our opinion, in the fact that the electrolytes used in preparation of metal iodate hydrosols are not completely dissociated salts,³⁶⁻⁴⁰ and that metal iodates form rather stable complexes in excess of one of the constituent ions.^{22, 41} These facts must be connected with the finding that higher order Tyndall spectra appeared only at the ionic solubility side of the precipitation curves of metal iodates,²² thus meeting the requirements of LaMer's theory for relatively small supersaturation needed to produce monodispersed colloid. The formation and the increase in concentration of molecularly dissolved, slightly dissociated complex [Pb (IO₃)₂(aq)] or [La (IO₃)₃(aq)] formed upon the mixing would correspond to the generation of molecularly dissolved sulphur in LaMer's experiments, leading finally to limiting state of supersaturation and outbursting of nuclei. The subsequent growth by diffusion of constituent ions onto nuclei assures the uniformity and the growth to particle sizes sufficiently

^{*} It ought to be remarked that Ginell et al.²⁰ noted in 1947 the appearance of higher order Tyndall spectra in silver chloride sols in $95^{0}/_{0}$ ethanol, which were prepared by direct mixing of precipitation components. We have tried several time to repeat the preparations they have described, but without success. Furthermore, one of us (J.K.) has investigated in a series of papers (cf. e.g., refs. 32 to 35) the precipitation and coagulation of silver chloride in water-ethanol mixtures (up to $90^{0}/_{0}$ of ethanol). The appearance of higher order Tyndall spectra has never been discerned. Unfortunately, the paper of Ginell et al. has been announced as the first communication in a series, but the subsequent papers did not appear as yet.

large that Tyndall spectra can be observed. Needless to say that it is a very simplified and uncomplete picture, but it is the best one we can offer at present. If it would be possible to resolve the nucleation stage more distinctly from growth stage (e.g., by change of temperature or solvent, or by generating iodate ions slowly by a chemical reaction in the presence of metal ions), the mechanism or formation of monodispersed metal iodates sols would be closer to its solving. Our efforts are now directed primarily toward this aim.

It is very difficult to say anything definitely about the causes of the formation of spherical particles in our systems. Berestneva and Kargin²¹ have investigated six typical colloidal systems (TiO₂, SiO₂, As₂S₃, Al(OH)₃, V₂O₅ and Au) by means of electron microscopy and electron diffraction. They established that the formation of colloidal particles go through two stages: initial formation of spherical or shapeless units of amorphous structure, which in process of ageing undergo crystallization in which these particles break up into more numerous but crystalline units. Since colloidal particles are formed from supersaturated solutions, the authors believed that the formation of spherical particles is most probable: the collisions of molecules, atoms or ions in supersaturated solutions are equally probable and frequent in all directions; so the primary formation of new phase must lead to spherical shape and amorphous structure. As the processes and reactions used in preparation of sols investigated by Berestneva and Kargin did not involve direct precipitation from electrolytic solutions, it is impossible to say if their explanation holds for our systems. It is necessary to know more about the structure of metal iodate particles before a conclusive answer can be given.

Owing to suitable optical properties, very simple preparation, spherical shape and convenient range of sizes, the possibility to use the monodispersed metal iodate hydrosols as extremely suitable standards for electron microscopy is very challenging. Moreover, the importance of monodispersed sols in studies of light scattering and stability of colloids cannot be underestimated. Thus, the continuation of this research seems to be highly warranted.

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IZVOD

Istraživanje monodisperznih hidrosolova metalnih jodata pomoću rasipanja svijetla i pomoću elektronske mikroskopije

M. J. Herak, J. Kratohvil, M. M. Herak i M. Wrischer

Pripravljeni su hidrosolovi lantanova i olovnoga jodata, čija su svojstva istraživana pomoću rasipanja svijetla, pomoću elektronske i obične mikroskopije, te mjerenjem brzine depozicije čestica. Solovi pokazuju izrazite i lijepe Tyndallove spektre, koji upućuju na nazočnost sferičnih čestica podjednake veličine. Solove olovnoga jodata, koji su vrlo nestabilni, moguće je stabilizirati filtriranjem kroz filtar-papir. Filtriranje je popraćeno promjenom naboja čestica. Rast čestica lantanova jodata mnogo je sporiji. Kutna raspodjela rasipanja svijetla u solovima metalnih jodata pokazuje tipične značajke monodisperznih sistema s česticama od nekoliko stotina milimikrona u polumjeru. Elektronska mikroskopija potvrdila je te rezultate. Polumjer čestica lantanova jodata iznosi oko 700 mµ, a polumjer čestica olovnoga jodata oko 860 mu. Varijacije veličine čestica u različnim uzorcima bile su znatne, a mogu se tumačiti nekontroliranim utjecajem izravnoga miješanja reakcionih otopina. Predložen je vjerojatni mehanizam nastajanja monodisperznih solova metalnih jodata na temelju LaMerove teorije o nastajanju monodisperznih koloida.

INSTITUT »RUĐER BOŠKOVIĆ« ZAVOD ZA FIZIKALNU BIOKEMIJU SKOLA NARODNOG ZDRAVLJA »ANDRIJA ŠTAMPAR« MEDICINSKI FAKULTET FIZIČKO-KEMIJSKI INSTITUT PRIRODOSLOVNO-MATEMATIČKI FAKULTET ZAGREB

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