

## Exploring the Detection Limits of Chemical Microscopy

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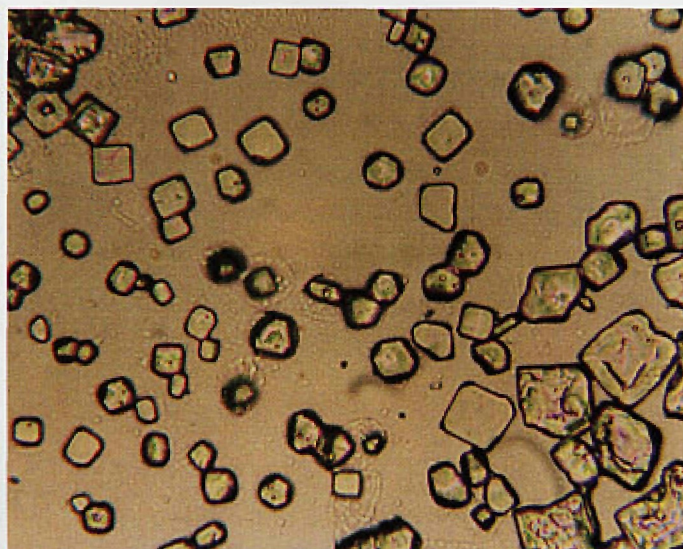
Chemical microscopy techniques were once a major source of information for scientists who wanted to detect the presence of metals in samples. Many microscopists used such techniques, which, when properly applied and appropriate checks for interferences made, could conclusively prove the presence of small quantities of specific metals.

The fact that the identification could be conclusive for trace amounts following relatively simple and cheap processes, and that visual evidence resulted, made these techniques quite robust. Chamot and Mason's text "Handbook of Chemical Microscopy" is held to be a definitive resource for these techniques<sup>1</sup>. With the development of absorption and emission based spectral analytical techniques, the application of chemical microscopy techniques dwindled, perhaps because the skills needed by analysts using spectral techniques were to be more rapidly acquired than those involving microscopy.

Our present study is a result of posing two questions:

- What are the limits of detection associated with microchemical techniques and can we corroborate the lower limit of detection by comparison with ICP?
- Could microchemical techniques become a simple, useful part of the rapid screening of environmental samples for traces of heavy metal contamination?

Reference samples of established concentrations of  $Pb^{2+}_{(aq)}$  were reacted on microscope slides with an equal volume of  $KI_{(aq)}$  of 0.01M concentration. The volume was reduced with gentle heating to about half that of the original. Crystallisation events were noted at the perimeter of the drops. On close examination at magnifications of 400 x we were able to record hexagonal  $PbI_{2(s)}$  at concentrations as low as 1.0 ppm, but irrespective of how many times we prepared specimens. All procedures were carried out at least six times and we could not find  $PbI_{2(s)}$  at any  $Pb^{2+}_{(aq)}$  concentrations lower than this. We prepared further test solutions which were of  $Pb^{2+}_{(aq)}$  concentrations of 990, 980 and 970 ppb. The concentrations of  $Pb^{2+}_{(aq)}$  used in this detection technique were confirmed using inductively coupled plasma (ICP) spectroscopy.

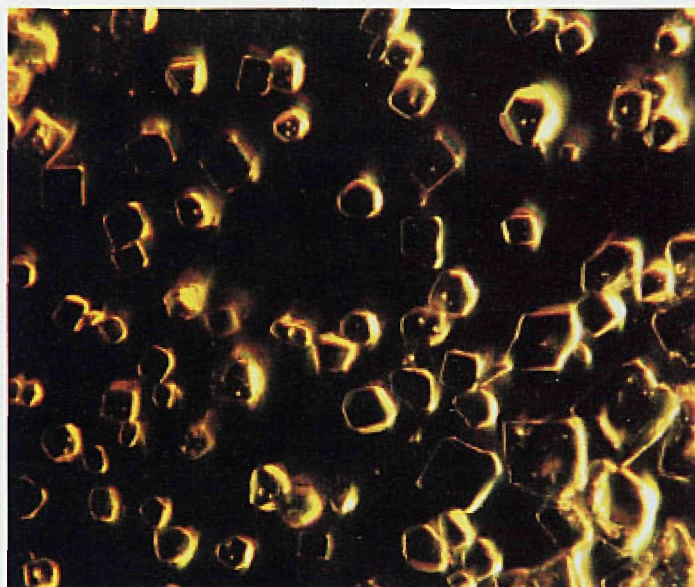


The first, a brightfield image taken using a polarising light shows several hexagonal micro-crystals of  $PbI_{2(s)}$  which were formed at  $Pb^{2+}_{(aq)}$  concentrations as low as 1.0 ppm (or 1 mg/L). Note that many of the hexagons are highly refractive and pleochroic.

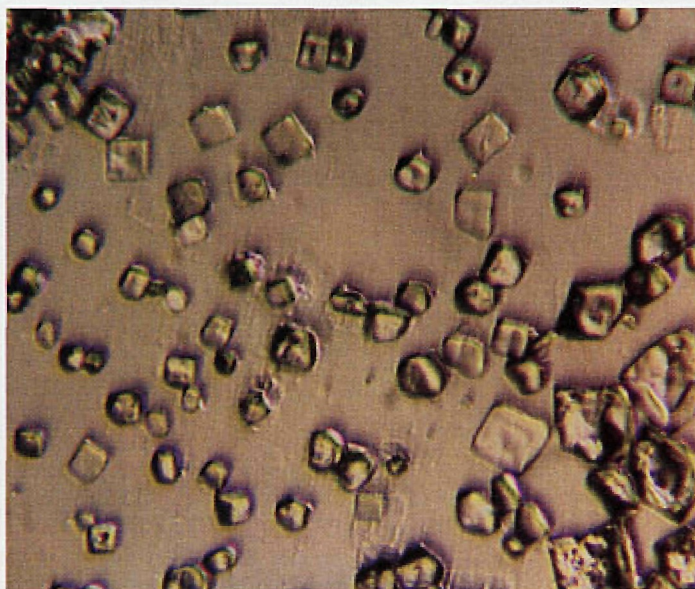
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It is evident that these microchemical techniques may be used to detect  $Pb^{2+}_{(aq)}$  at concentrations as low as 1.0 ppm, and that such measurements may be made quickly. Further work in our laboratory will explore other methods for  $Pb^{2+}_{(aq)}$  such as the triple nitrite reaction, the influence of other ions on the minimum limit of detection, and also what the minimum limit of detection for  $Hg^{2+}_{(aq)}$  will be. Our work to date suggests that this latter ion may be detected at concentrations as low as 600 ppb. All of our work to date suggests that these chemical microscopy techniques are simple to make, robust, and may well contribute to the rapid screening of environmental samples. They would seem to be well suited to screening processes remote from sophisticated heavy analytical equipment. ■

1. Chamot, E.M. and Mason, A.B. (1989) Handbook of Chemical Microscopy, vol. II, McCrone Research Institute, Chicago, IL.



The second is a darkfield version of the same visual field. The hexagonal crystals are scattering light in such a way to suggest increased crystal growth above the plane of focus, unlike the  $KI_{(s)}$  counterparts which are commonly square in cross-sections.



The third micrograph was taken using Hofmann Modulation Contrast (HMC) and the three dimensional difference between  $PbI_{2(s)}$  and  $KI_{(s)}$  is reinforced.





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