Direct Compressibility Measurements of Liquids up to 7 Kilobar Using Pressure Generator with Integrated Piston Position Sensor.

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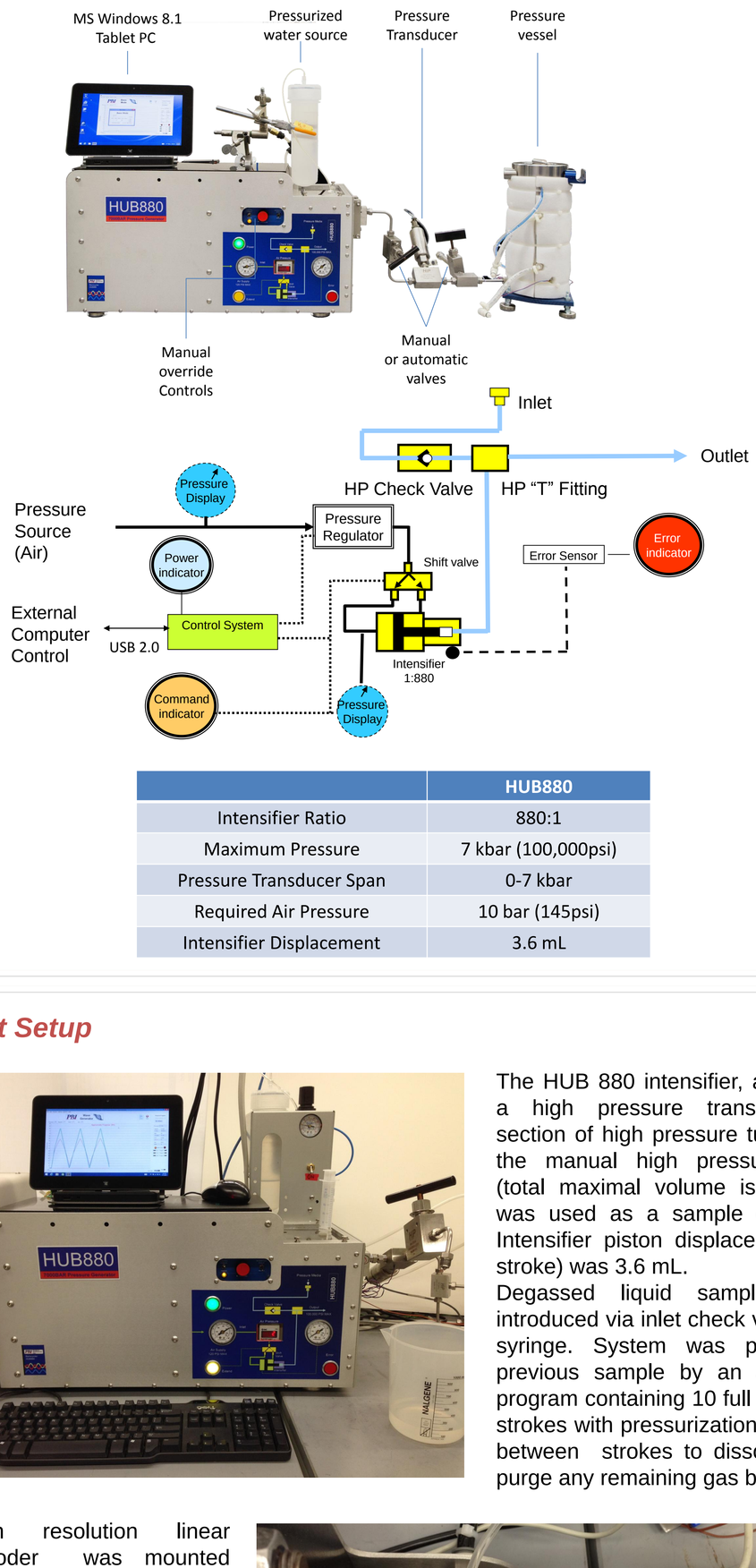
Abstract

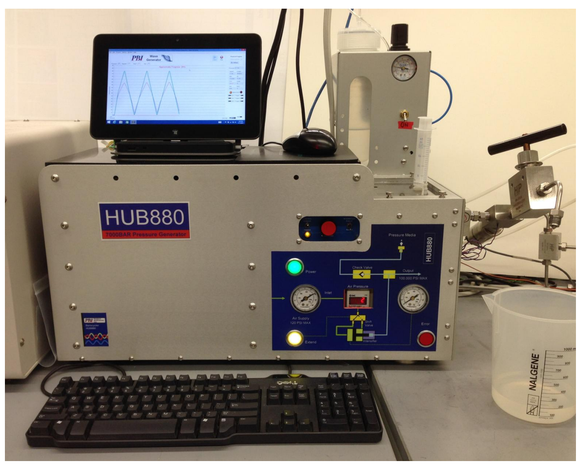
High pressure in the range of hundred to thousand bars is now recognized to have fundamental effects on the structure and function of biological systems from proteins, cell, to whole organisms. Much of the high pressure equipment in use for biological applications, however, continues to be a domain for do-it-yourself enthusiasts. As a result, basic research and understanding of mechanisms governing high-pressure unfolding of proteins, pathogen inactivation, membrane fluidity, protein dynamics and other significant effects of pressure is progressing at a limited pace; the successful industrial applications of pressure in food and biopharmaceutical industry to date remain driven by empirical optimization efforts that are lengthy and costly. Commercialization of the HUB-series pressure generators opened up new opportunities in high pressure research, particularly in studies of protein dynamics and high pressure enzymology. Modular instrument design enables fast re-configuration of the instrument to drive pressure vessels [1], optical or magnetic resonance cells [2 - 4]. Moreover, a recent addition of the piston position sensor to the HUB880 pressure generator offers direct measurements of the sample compressibility (bulk modulus) and monitoring of phase transitions at pressure up to 7 kilobar using this instrument platform without a need for additional analytical equipment. This presentation will describe the principle behind direct compressibility measurements on a HUB880 platform and show examples of bulk modulus determination for several liquids. The rapid and precise bulk modulus measurements has sufficient resolution to determine the water content in extra virgin olive oil, predict flow rate fluctuations in gradient UPLC applications and potentially better understand pathogen inactivation using High Pressure Processing.

HUB880 Pressure Generator: Flow Diagram and Specifications



	HUB880
Intensifier Ratio	880:1
Maximum Pressure	7 kbar (100,000psi)
Pressure Transducer Span	0-7 kbar
Required Air Pressure	10 bar (145psi)
Intensifier Displacement	3.6 mL

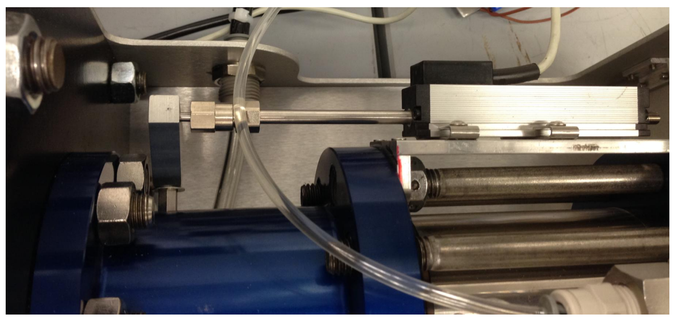
Test Setup



The HUB 880 intensifier, a tee with a high pressure transducer, a section of high pressure tubing and the manual high pressure valve (total maximal volume is 5.1 mL) was used as a sample container. Intensifier piston displacement (full stroke) was 3.6 mL.

Degassed liquid samples were introduced via inlet check valve by a syringe. System was purged of previous sample by an automatic program containing 10 full intensifier strokes with pressurization to 1 kbar between strokes to dissolve and purge any remaining gas bubbles.

High resolution linear encoder was mounted directly onto an intensifier and coupled with a lever connected to an intensifier plunger between air and water seals. Signal from this piston position sensor was acquired by the auxiliary ADC channel on the HUB880 control board.

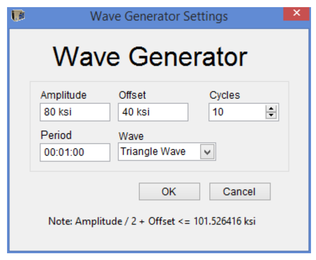


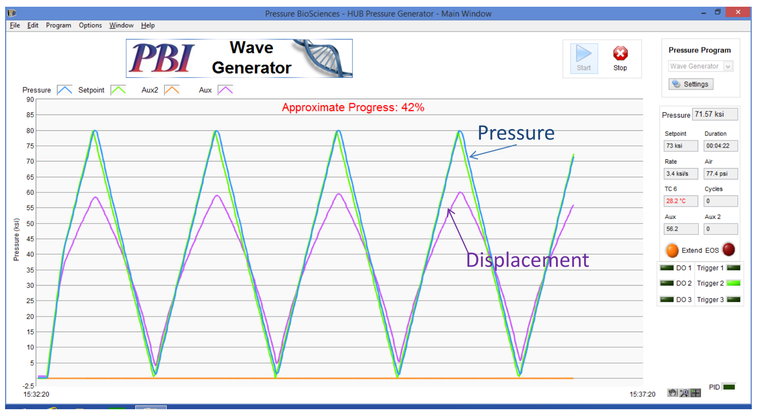
Conclusions

Compressibility data for various fluids are currently incomplete and only available for selected temperature values [5-7]. Instrument described here offers an ability to acquire such data in automated fashion by manipulation of pressure and temperature and recording corresponding volumetric displacement. Correction for the seal friction can be derived form the acquired data and experimental values of bulk modulus can be obtained. Our data suggests that the system provides enough resolution and accuracy to match published data, when available. The built-in software temperature control and optional resistive heating blanket can be used to automatically acquire compressibility date for a broad range of temperatures without reloading the sample. Better understanding of compressibility of aqueous solutions, organic solvents and their mixtures with water may provide useful information for development of improved HPP processes for juices and alcoholic beverages. Moreover, such data may help to further improve instrumentation and consumables for ultra high pressure liquid chromatography (UHPLC), as well as many more useful applications.

Automatic Data Collection

The HUB880 was configured to output pressure from 0 to 80,000 psi (5.516 kilobar) following a slow triangular waveform to allow for dissipation of adiabatic heat into the metal components of the system. Isentropic experiments can be conducted using rapid pressure cycling.





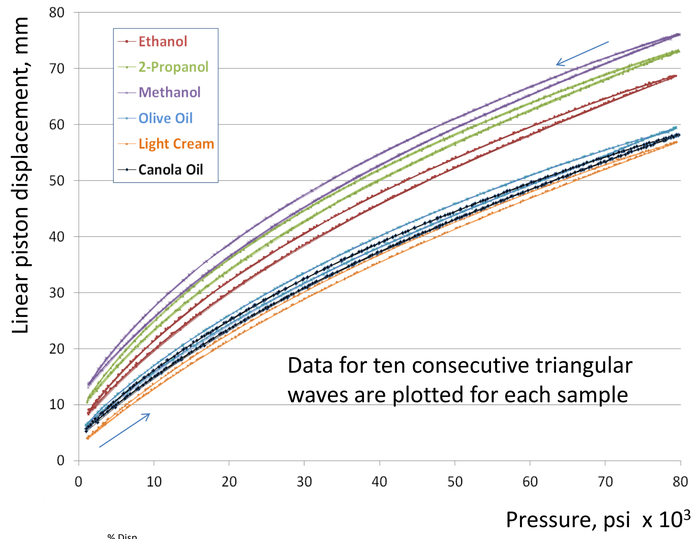
Piston position sensor data were recorded automatically by a control system along with pressure and temperature values. Data were stored as MS Windows ® .CSV (comma-delimited file) format and analyzed in MS Excel.

Isothermal Compressibility and Bulk Modulus of Liquid Samples

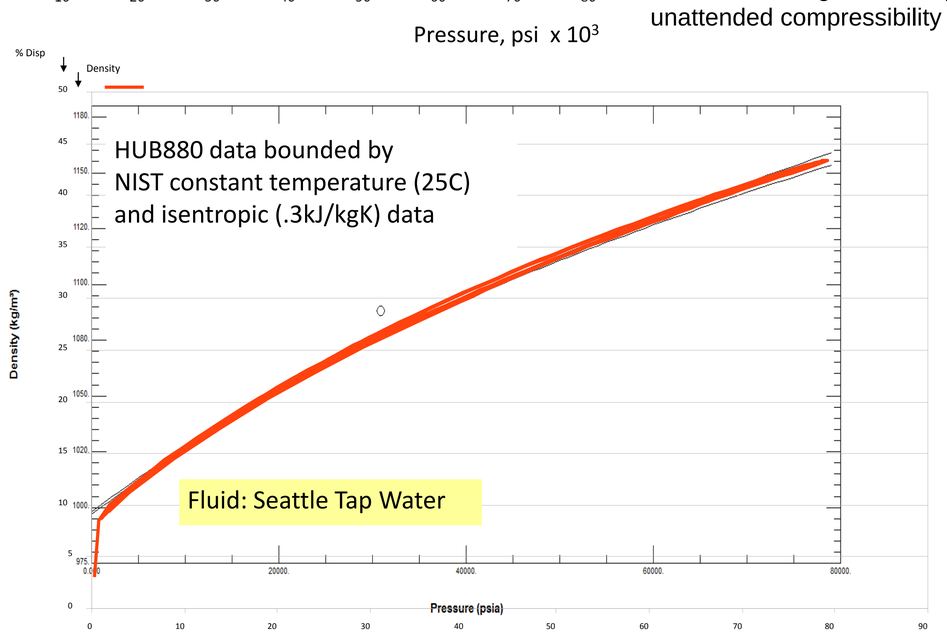
The pressure dependence of volume is described to first order by the isothermal compressibility coefficient κ defined as

$$\kappa_T = -(1/V)\,(\partial V/\partial P)_T$$

where *V* is the volume and *κ* is bulk modulus at constant temperature T.



Isotherms shown here are plotted as linear piston displacement versus pressure in thousands of psi. Since diameter of the piston (9.525 mm) remains constant, linear displacement in a leak-free system is directly proportional to the volume change and, therefore, ΔV can be derived. The system records both pressure and volume change values automatically over a wide range of pressure values. Resulting data contain sufficient information to calculate bulk modulus of the sample at given temperature. The HUB880 instrument is capable of programmable temperature control, offering an ability of complex unattended compressibility tests.



HUB880 data appear superimposed onto the isothermal and isentropic water compressibility curves. The NIST water compressibility data were used as a normalization standard for other sample types.

Sample	κ, Experimental Value at 23°C, (MPa^{-1})	κ, Literature Value (Mpa^{-1} at 20°C) [5-7]
Water	4.55	4.591
Ethanol	11.28	11.19
Methanol	14.81*	12.14
2-Propanol	13.45	13.32
Olive Oil	5.87	n/a
Canola Oil	5.16	n/a

* Possible evaporation inside intensifier during sample aspiration resulting in higher than expected compressibility

References

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