TABLE 5

Anvil-type pressure cells (solids) used for optical spectroscopy [a]

Pressure limits (kbar) [b]	Optical Instrument	Windows	Wavelength range (μm)	Remarks	Ref.
100	Perkin-Elmer models 421, 350, Beckman IR-4	Diamond [c,d]	2—35	6 × beam condenser	48
200	Perkin-Elmer models 225, 350, FTS-14	Diamond	0.27—40	6 × beam condenser	62, 63
100	Perkin-Elmer model 301	Diamond	16—200	6 × beam condenser	64
100	Beckman IR-11	Diamond	16—200	6 × beam condenser	65
100	Beckman IR-12	Diamond [d]	2.5—16	6 × beam condenser	65
100	Cary-14	Diamond	0.25—2.5	With or without a beam condenser	66
100	Digilab FTS 20 A	Diamond	1—1000	With beam condenser	68
100	Beckman FS-520 interferometer	Diamond	FIR → 250	Light pipe necessary	67
100	Beckman #4260	Diamond [d]	2—50	With beam condenser	69
35	Michelson Interferometer	Quartz		Cube-anvil type cell	70
50 [f]	(?)	Diamond	NIR	Cube-anvil type cell; sample contained in NaCl; uses quartz or sapphire for lower pressures	71
150	FTS-14	Diamond	2.5—200	298—673 K	e(1)
200	PE 421, 210, Cary 14R, Spex 1700	Diamond	0.2—40	To 973 K	e(2)
	FTS-14	Diamond	2.5—500		e(3)
10	Cary 14R	Diamond	0.19—5	123—623 K	e(4)
100	Perkin-Elmer model 225	Diamond		Refracting beam condenser	73

[a] DAC can be used for liquid or solutions if a gasket is used between the anvils. [b] DAC can be used routinely to 100 kbar if diamonds are properly aligned. [c] Diamond-Type I has absorptions at 3 μm (weak), 4—5.5 μm (intense), 7—10 μm (intense); Type II has absorptions at 3 μm (weak), 4—5.5 μm (intense). [d] Sapphire windows may be used from 2—5 μm. [e] Personal communication: (1) R.J. Jakobsen, Battelle Memorial Inst.; (2) G.J. Piermarini, Nat. Bur. Stand.; (3) G. Carlson, Westinghouse, Pittsburgh, Pa.; (4) C.A. Angell, Purdue Univ. [f] Claimed to be used to 1273 K.

0.037 in.) a means of condensing the source beam becomes necessary. In the case of a grating double beam spectrophotometer a beam condensation process is used (usually from 4—6X condensation) [48,62—66]. In the case of interferometric measurements a light-pipe has been used both for the entrance energy and the exit energy in the far IR region [67]. Recently, a new Harrick 6X beam condenser has been interfaced with the Digilab 20A interferometer [68]. A 4X beam condenser has also been coupled with a Beckman Model no. 4260 [69].

Using diamonds as the optical windows puts a stringent requirement on the properties of the beam condenser. The critical angle of diamonds is 26°. As a result the beam condenser should condense the light in a cone having an angle less than 52°. Also, since the faces of the diamonds are so small, the spot size of the condensed light should be as small as feasible, 1 mm^2 or less. One can then be sure that most of the condensed light is reaching the sample between the two diamonds and is not clipped by the large size of the DAC. Adams and Sharma [73] have recently reported a refracting beam condenser for IR use with the DAC. The optical problems associated with IR spectroscopy with a DAC are discussed [73].

For Raman scattering experiments both the DAC and the piston-cylinder may be used without any condensation of source energy, since lasers are used as the exciting sources with narrow beam radii. The laser light can be focussed directly into the small optical aperture of the pressure cell without too much difficulty.

(iii) Instrumentation for Raman spectroscopy at high pressure

Both the piston-cylinder and anvil-type cells have been used for obtaining Raman spectra at high pressure. Although energy problems are severe in IR spectroscopy because of the small optical aperture in the high pressure cells, the advent of laser sources for Raman spectroscopy has overcome a great deal of these problems.

The first Raman spectra obtained in a DAC were made in 1968 [59,60]. The red → yellow transition in HgI_2 followed. In both cases 0° scattering geometry was used, although it was cited that 180° scattering could be used [59]. The DAC was also used for the study of liquid Br_2 and CS_2 [61]. However, except for solids with high Raman scattering efficiencies, the final results were disappointing. Recently, the DAC has been used with certain modifications and with improved results [53]. Adams et al. used a tungsten carbide window in place of one of the diamonds in the DAC [53]. The details are illustrated in Fig. 6. Both 90° and 180° scattering geometries were used. One limitation is that the tungsten carbide limits the maximal pressures to ca. 30 kbar. The tungsten carbide allows the Raman intensity to build up by allowing the excitation energy to traverse twice through the sample and additionally acts as a mirror. It has been suggested that back-silvering of one diamond anvil would give comparable results in the DAC and allow the DAC to be used