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Developments and Trends in Flow Injection Atomic Absorption Spectrometry

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An overview is given on the development of flow injection atomic absorption spectrometry (FI-AAS) within the last decade, highlighting the main achievements and trends in the field within the period. The review is appended by a full bibliography covering the period from 1972 to early 1995, indexed according to the sub-disciplines of AAS, techniques, application fields and analyte species.

Keywords: *Atomic absorption spectrometry; flow injection*

As early as 1972, Winefordner's group[452] proposed a sample introduction system for flame atomic absorption spectrometry (FAAS), which today may be rightfully designated as flow injection (FI). However, except for two more contributions, the technique did not seem to have aroused further interest before the foundations of flow injection analysis (FIA) were laid following the pioneering work of Růžička and Hansen. The first reports on techniques explicitly defined as FI-AAS were published in 1979.[598,616] The rapid increase of publications thereafter is shown in Fig. 1. Originally mostly considered to be merely an alternative, although perhaps more efficient, means of sample introduction for FAAS, FI was soon found to be an ideal tool for manipulating chemical reactions and other sample pre-treatment procedures associated with AAS measurements. In this respect, the contributions from Astrom[15] in 1982 and Olsen *et al.*[395] in 1983 are amongst those that deserve special mention. The decade (1984–1994) which followed was one in which the rapid progress made in the combined technique intensively stimulated the development of both AAS and FIA in general. The development is now marked by the accumulation of more than 600 publications up to the end of 1994, and two dedicated monographs, one in 1989, edited by Burguera,[72] and another by one of the present authors (Fang),[194] published last September.

The distribution of publications in the various sub-disciplines of AAS is shown in Fig. 2. Flame photometry and atomic fluorescence applications are also included in the estimation. Flow injection inductively coupled plasma emission spectrometry (ICP-ES) and mass spectrometry (ICP-MS), which deserve separate treatment, are not included in this review. Publication distribution in the various FI-AAS techniques and application fields is shown in Figs. 3 and 4, respectively. The strong interest in on-line separation and preconcentration systems is well illustrated by over 200 contributions in this field, which constitutes about one third of the total number of publications in FI-AAS and half of the total number dealing with FI techniques for AAS.

* Published in celebration of the Tenth Anniversary.

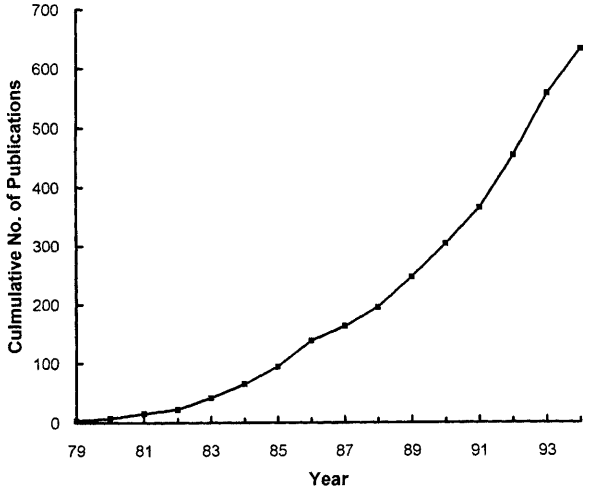

Fig. 1 Culmulative number of publications on FI-AAS

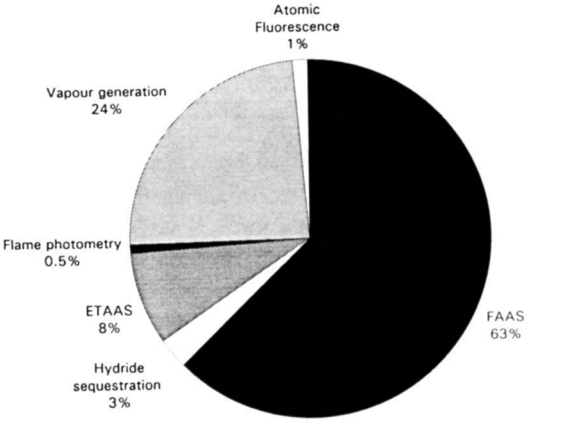

Fig. 2 Distribution of publications in various sub-disciplines of AAS

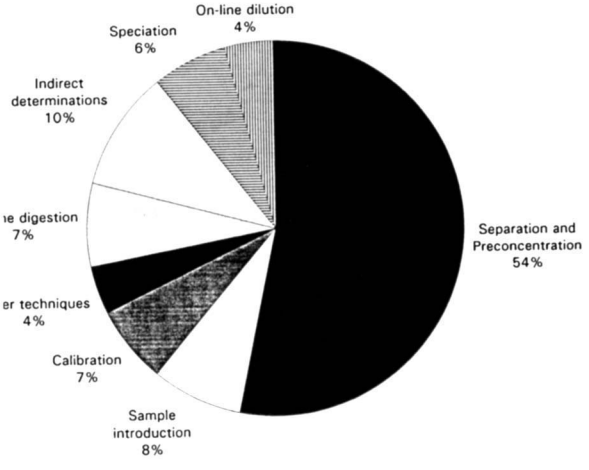

Fig. 3 Distribution of publications in FI-AAS techniques

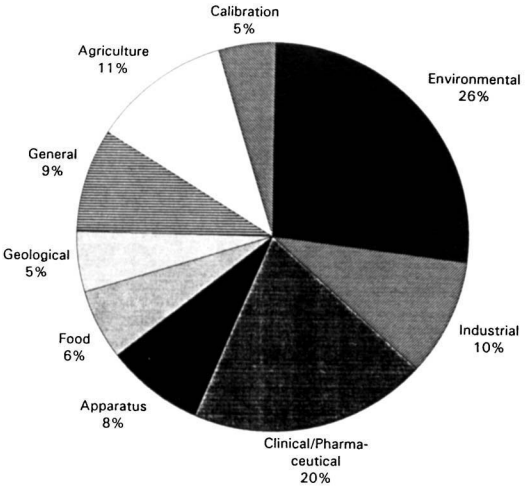

Fig. 4 Distribution of publications in various application fields

The main developments in the past decade and recent trends in the various FI-AAS techniques are highlighted in the following sections. A complete bibliography on FI-AAS covering the period from 1972 to early 1995 is appended. The entries are indexed according to the technique fields, area of application, and analyte species in Tables 1, 2 and 3, respectively.

FI SAMPLE INTRODUCTION FOR FAAS

Despite some early suspicions, the general suitability of the flame AAS detector for direct connection with FI sample introduction now appears to be well-accepted. Except in cases where sample volumes are extremely small, the relatively large capacity of the spray chamber does not contribute significantly to dispersion of the sample, and the kinetic features of a typical AA detector were found to provide an almost undistorted picture of the injection process.[180] However, high resolution readout systems with time constants in the 0.02–0.1 s range are required to reflect the response faithfully. Under such conditions, the dispersion contribution of a typical FAAS detector was shown to be merely equivalent to that of a 10 cm length of 0.5 mm id tubing with only 20 µl volume, and 90% steady-state signals can be achieved with as little as 45 µl of sample.[180]

Noteworthy observations related to the FI mode of sample introduction for FAAS within this period include the following:

(a) Except at extremely low carrier flow rates of below 2 ml min^{-1}, or with wide peaks (*e.g.*, over 20 s for baseline-to-baseline recording), peak height evaluation usually produces better precision than peak area evaluation.[180]

(b) Under typical FI conditions viscosity and temperature variations in the sample do not significantly affect FAAS signals.[531]

(c) Compensation of starved sample flow (by introducing air or water through a T-piece) rarely, if ever, improves the performance of FI-FAAS systems, while de-gassing of the carrier solution is always beneficial under flow-starvation conditions.[174]

(d) The high tolerance to samples with high dissolved solids contents of the FI mode of sample introduction is now well-recognized.[175,360] This is well-demonstrated by the repetitive introduction of a saturated lithium borate solution for 4 h (465 injections) with a relative standard deviation of 1.5%,[175] and repetitive introduction of a 30% m/v NaCl solution for 1.5 h (250 injections).[180]

(e) A recent trend is the extension of the latter capability to the direct introduction of slurry samples, including sewage,[333]

Table 1 Methods and techniques

Method/technique	Detection*	References
Separation and preconcentration:		
Ion exchange	F	22, 42, 65, 91, 92, 93, 115, 134, 117, 118, 139, 149, 159, 164, 165, 166, 172, 173, 176, 181, 188, 242, 243, 244, 246, 247, 248, 252, 259, 265, 266, 272, 294, 306, 326, 330, 356, 357, 359, 361, 362, 363, 399, 401, 403, 405, 413, 414, 415, 416, 419, 443, 465, 508, 509, 510, 514, 553, 602, 615, 625
	ET	22, 34, 136, 378, 499, 613
	HG	97, 188, 263, 381, 600, 605, 634
	CV	633
Sorbent extraction	F	23, 35, 58, 96, 111, 147, 181, 187, 193, 217, 227, 262, 271, 275, 277, 283, 289, 307, 308, 319, 327, 328, 373, 376, 377, 393, 405, 418, 421, 437, 442, 449, 490, 509, 554, 584, 588, 603, 609, 621, 635
	ET	44, 136, 179, 289, 307, 320, 409, 476, 477, 478, 479, 481, 484, 564, 579, 582
	CV	217
Liquid–liquid extraction	F	112, 114, 132, 143, 206, 208, 209, 251, 258, 261, 278, 280, 317, 344, 351, 352, 369, 385, 386, 390, 448, 469, 488, 489, 495, 500, 639
	ET	25, 26, 27, 41, 119, 320
Coprecipitation	F	141, 183, 184, 400, 581
	ET	138, 189, 638
	HG	498
Precipitation	F	2, 3, 130, 144, 145, 146, 147, 148, 153, 156, 157, 159, 160, 161, 210, 281, 284, 340, 341, 342, 343, 353, 363, 365, 404, 446, 447, 548, 550, 645
In situ collection of hydride/mercury	ET	6, 321, 322, 323, 324, 346, 468, 471, 472, 473, 496, 499, 516, 571, 597, 630, 644
Dialysis	F	273, 296, 297, 417, 555
Separation of interferences	F	130, 265, 326, 338, 355, 488, 549
	ET	50
	HG	255, 263, 388, 389, 543
Electrochemical	F	37
	ET	40
	HG	60, 302
Slotted tube atom trap	F	190, 214, 505, 606, 609
Organic solvent effect	F	17, 20, 21, 190, 205, 576
On-line dilution	F	39, 100, 126, 187, 192, 216, 312, 424, 425, 526, 531, 534, 535, 538, 558, 608
On-line pre-reduction	HG	197, 407, 451, 485, 587, 607
	CV	492, 493
Indirect determination	F	108, 109, 110, 144, 155, 156, 157, 203, 208, 209, 210, 218, 230, 235, 261, 262, 282, 284, 335, 336, 337, 338, 339, 342, 343, 344, 364, 365, 366, 367, 368, 369, 404, 448, 469, 548, 552, 614, 645

Table 1 (*continued*)

Method/technique	Detection*	References
On-line digestion	F	76, 79, 81, 83, 99, 103, 125, 128, 160, 220, 221, 226, 233, 239, 345, 617, 618, 619
	ET	77
	HG	16, 95, 314, 485, 515, 587
	CV	162, 515, 517, 518, 583
Calibration	F	8, 11, 89, 90, 167, 168, 213, 249, 276, 310, 398, 475, 519, 521, 522, 525, 526, 527, 529, 531, 534, 535, 536, 538, 623
	HG	331
Sample introduction:		
General	F	62, 63, 64, 94, 116, 119, 274, 333, 422, 423, 474, 520, 523, 539, 551, 624
High-pressure nebulization	F	45, 46, 47, 50, 336, 411, 412, 454, 573
Microsampling	F	19, 214, 452, 457, 466
Thermospray	F	260, 274, 285
	ET	29, 30
Speciation	F	43, 48, 57, 68, 148, 314, 318, 349, 357, 358, 363, 377, 399, 412, 491
	ET	58, 478, 479, 481
	HG	85, 123, 124, 197, 354, 407, 440, 575
	CV	55
Reviews		28, 66, 102, 109, 110, 127, 137, 163, 170, 171, 177, 178, 186, 191, 207, 212, 214, 250, 251, 281, 303, 316, 317, 330, 355, 360, 392, 402, 428, 429, 430, 436, 441, 466, 511, 512, 519, 530, 531, 541

* F = Flame; ET = electrothermal; HG = hydride generation; and CV = cold vapour.

pigments,[311] calcinated vegetables,[561] silicates[313] and homogenized food samples.[122]

In addition to the conventional pneumatic nebulization system, FI was also found to be the sample introduction mode of choice for thermospray[285,430] and high-pressure nebulization systems introduced more recently.[45,49,50] Significant improvements in sensitivity can be obtained with these systems. However, special nebulization equipment and high-pressure HPLC pumps are required for their operation.

FI SAMPLE INTRODUCTION FOR VAPOUR GENERATION (VG) AAS

The FI mode of sample introduction for performing hydride generation and cold vapour generation AAS has undergone rapid development since the publication of Astrom's pioneering work in 1982.[15] This development is now marked by a total of 145 related publications, estimated from the appended bibliography, and several commercialized models of dedicated equipment for FI-VGAAS. Owing to the obvious and multiple advantages gained over conventional batch operated (including automated) systems, it appears to be only a matter of time before FI will be recognized and practised as the standard mode of operation for VGAAS. In addition to the already well known advantages of high sample throughput, low sample/reagent consumption, and good reproducibility, possibilities of further improving selectivity using FI techniques have aroused intense interest. The in-depth exploitation of kinetic discrimination effects, the implementation of on-line matrix separation or matrix modification represent current efforts for achieving

Table 2 References listed according to area of application

Area of application	References
Agricultural:	
Fertilizers	639
Pesticides	261, 262, 335
Plant materials	236, 240, 370, 443, 451, 496, 531, 565, 566, 567, 568, 581, 610, 611, 612, 621, 630, 632
Grains	111, 122, 168, 193, 560, 567, 568, 571
Vegetables	105, 122, 469, 496, 561
Environmental:	43, 88, 168, 222, 320, 322, 333, 347, 496, 565, 566, 644
Waters	5, 31, 47, 48, 55, 91, 93, 96, 105, 111, 126, 140, 164, 165, 166, 172, 176, 181, 209, 233, 247, 252, 258, 272, 341, 343, 350, 352, 362, 381, 408, 420, 437, 443, 450, 471, 476, 477, 481, 482, 487, 496, 498, 499, 559, 568, 575, 580, 583, 600, 601, 602, 605, 610, 613, 633, 634, 635, 637, 642
Sea-waters	22, 123, 165, 179, 181, 217, 259, 307, 315, 320, 363, 378, 394, 395, 409, 478, 554, 564, 580, 605, 612, 620, 634, 638
Air	198, 199, 200
Coal fly ash	168, 313, 319, 567, 610, 611
Soils	105, 124, 150, 168, 171, 298, 319, 453, 557, 568, 580, 600, 601, 631, 632, 634
Waste materials	150
Industrial:	215, 254, 434, 501, 636
Ceramics	120, 179
Lubricating oils	56, 439
Metallurgical	421
Metals and alloys	51, 52, 60, 231, 294, 323, 347, 384, 388, 389, 460, 467, 500, 505, 525, 543, 563, 586, 592, 610, 617, 618, 619, 624, 630, 641
Cement	32, 334, 445
Other	87, 204, 410, 553, 588, 629
Clinical:	
Animal tissues	33, 77, 83, 212, 236, 319, 405, 406, 581, 630
Hair	193, 627
Blood/plasma	75, 79, 84, 138, 211, 226, 290, 292, 293, 348, 387, 580, 581, 587
Serum	4, 9, 10, 18, 67, 73, 113, 206, 243, 244, 431, 432, 433, 435, 466, 470, 494, 499, 547, 574, 628, 643
Urine	9, 70, 84, 139, 206, 225, 233, 234, 263, 266, 287, 288, 365, 375, 485, 580, 581, 583
Other body fluids	69, 71, 78, 80, 243, 382
Pharmaceutical	96, 142, 143, 144, 145, 218, 284, 364, 365, 366, 367, 368, 369, 448, 496, 552, 626
Food:	33, 188, 220, 374, 560, 562, 640
Fruits	74, 122, 212
Edible oils	101
Beer and wine	23, 28, 95, 295, 296, 297
Beverages	12, 13, 28, 95, 443
Milk	14, 444, 555, 556
Biological materials	76, 77, 125, 168, 183, 193, 202, 203, 222, 245, 304, 320, 322, 405, 581, 604
Geological/mineralogical	3, 104, 107, 213, 227, 268, 269, 299, 313, 353, 380, 391, 400, 401, 417, 421, 446, 447, 450, 495, 499, 603, 609, 623
General	1, 2, 3, 7, 36, 38, 61, 98, 177, 180, 196, 200, 219, 238, 267, 270, 372, 383, 392, 425, 458, 459, 461, 480, 483, 524, 528, 531, 537, 542, 544, 545, 546, 590, 593, 594, 595, 616
Apparatus	24, 26, 27, 59, 133, 192, 228, 232, 237, 245, 253, 257, 264, 285, 291, 300, 301, 302, 306, 321, 325, 329, 331, 397, 398, 427, 504, 507, 508, 510, 540, 599, 610, 612, 625
Theory	7, 38, 54, 98, 174, 180, 185, 238, 267, 530
Reviews	168, 428, 429, 434, 436, 441, 474, 497, 508, 511, 512, 515, 519, 530, 541, 551, 570, 584

such goals. The determination of hydride-forming elements in copper- and nickel-based alloys by Tyson's group[389,543] is a good example of the efficacy of such systems.

Although still at an early stage of development, the application of FI sample introduction in vapour generation atomic fluorescence spectrometry (AFS) was rewarded with an even

Table 3 Species determined

Species	Detection* and FI contribution	References
Alkaloids	F, indirect, with liquid–liquid extraction	143
Aluminium	F	43, 260, 339, 408
	with ion exchange	243, 244, 308, 361, 362, 403, 443
	with thermospray	260
	with dilution	100
	ET	12, 14
Ammonia	F, indirect	155
Arsenic	HG	16, 52, 58, 85, 88, 105, 140, 150, 198, 214, 222, 224, 231, 234, 256, 287, 288, 315, 350, 374, 375, 380, 440, 451, 453, 456, 485, 503, 506, 515, 543, 557, 559, 562, 568, 572, 587, 591, 592, 596, 607, 611
	with ion exchange	263, 381
	by electrochemical generation	60, 302
	ET	86, 478
	with *in situ* collection	6, 571, 644
	with thermospray	30
Antimony	F	641
	HG	52, 97, 123, 124, 224, 315, 420, 471, 515, 591, 592
	with ion exchange	605
	by electrochemical generation	302
	ET, with *in situ* collection	571
Beryllium	ET, with ion exchange	136
Bismuth	F	641
	HG	15, 51, 52, 106, 107, 224, 384, 420, 515
	with ion exchange	634
	ET, with *in situ* collection	571
Bromide	F, indirect, with precipitation	157
Bromazepam	F, indirect, with ion exchange	448
Cadmium	F	33, 49, 83, 139, 141, 150, 159, 181, 193, 245, 246, 266, 319, 370, 405, 414, 419, 514, 554, 581, 639
	with ion exchange	134, 149, 165, 166, 176, 248, 326, 394, 395, 615
	with sorbent extraction	147, 327, 604
	with high-pressure nebulization	46
	with electrolytic deposition	37
	ET	138, 189, 320, 476
	with liquid–liquid extraction	25, 27, 300
	with sorbent extraction	44, 307, 409, 564
	with thermospray	30
Carbonate	F, indirect	156, 161
Calcium	F	1, 2, 3, 5, 8, 10, 31, 32, 56, 69, 73, 74, 100, 113, 126, 129, 167, 211, 269, 426, 435, 444, 466, 547, 556, 563, 574, 622, 628, 629, 642, 643
	with ion exchange	265
	with sorbent extraction	23
	with precipitation	153
	with dialysis	296, 297, 555
	with slurry introduction	313, 560, 561
Cerium	F, indirect	338
Chloride	F, indirect, with precipitation	156, 343
	F, indirect, with dialysis	555
Chromium	F	48, 203, 260, 311, 357, 377, 412, 465, 482, 490, 524, 576, 624
	with ion exchange	248, 615
	with high-pressure nebulization	46
	ET, speciation, with sorbent extraction	481
Cobalt	F	4, 290
	with ion exchange	252, 326, 553, 602, 615
	with sorbent extraction	23, 147, 513
	with coprecipitation	141, 447, 581
	with thermospray	260
	with high-pressure nebulization	46, 49
	ET	
	with liquid–liquid extraction	25, 27
	with sorbent extraction	409, 477, 621
	with thermospray	30
	with high-pressure flow	50
Copper	F	4, 17, 20, 69, 71, 80, 82, 101, 213, 240, 278, 311, 457, 466, 631
	with ion exchange	42, 114, 118, 134, 149, 165, 166, 176, 242, 248, 252, 326, 356, 413, 419, 615

Table 3 (*continued*)

Species	Detection* and FI contribution	References
	with sorbent extraction	23, 35, 111, 147, 277, 283, 319, 327, 373, 376, 442, 449, 554, 604
	with liquid–liquid extraction	351, 352
	with precipitation	159, 446
	with electrolytic deposition	349
	with on-line digestion	76, 79, 81, 617, 618
	with on-line dilution	216, 431
	with high-pressure nebulization	49
	with slurry sampling	311
	ET	
	with ion exchange	613
	with liquid–liquid extraction	25, 26, 27
	with sorbent extraction	44, 307, 320, 409, 476, 564
	with coprecipitation	638
	with high-pressure flow	50
Cyanide	F, indirect, with precipitation	155, 160, 230
Diethyldithioformate	F, indirect, with liquid–liquid extraction	261
Dichromate	F, indirect, with precipitation	161
Dimethoxy-dithiophosphate	F, indirect, with liquid–liquid extraction	262
EDTA	F, indirect, with ion exchange	359
Gallium	F, with ion exchange	362
Germanium	ET	236
	with *in situ* hydride collection	324, 496
Glycine	F, indirect	218, 336
Gold	F	
	with ion exchange	134
	with sorbent extraction	227, 418, 421, 603, 609
	with liquid–liquid extraction	500
	with dialysis	417
	ET, with liquid–liquid extraction	300
Indium	F	
	with ion exchange	362
	with liquid–liquid extraction	114
Iodide	F, indirect, precipitation	157, 342
Iron	F	4, 68, 69, 71, 80, 82, 101, 122, 129, 240, 241, 275, 358, 433, 445, 629, 631, 636
	with ion exchange	248, 615
	with sorbent extraction	275, 327
	with precipitation	153
	with slurry sampling	32, 311, 313, 560, 561
	with on-line dilution	100
	with high-pressure nebulization	46, 49
	with electrolytic deposition	37
	with on-line digestion	76, 81
	ET, with liquid–liquid extraction	25, 27
Isoniazid	F, indirect, with liquid extraction	282
Lanthanum	F, indirect	338
Lead	F	32, 33, 57, 77, 93, 150, 213, 214, 242, 254, 387, 452, 501, 641
	with ion exchange	134, 149, 165, 166, 176, 201, 242, 248, 394, 395, 415, 419, 615
	with sorbent extraction	23, 91, 92, 117, 132, 147, 181, 283, 294, 319, 327, 437, 442, 554, 635
	with liquid–liquid extraction	70, 487
	with coprecipitation and precipitation	183, 341
	with slotted tube	505
	with electrolytic deposition	37
	with high-pressure flow	462
	with microsampling	452
	speciation	57
	ET	
	with ion exchange	34
	with liquid–liquid extraction	25, 27
	with sorbent extraction	44, 179, 320, 410, 476
	with coprecipitation	638
	with *in situ* collection	151, 597
	with on-line digestion	77
	with thermospray	30
	with high-pressure flow	50
	HG	152, 215, 304, 577, 640
	with on-line digestion	95, 515
Levamisole	F, indirect, with precipitation	284
Lithium	F	167, 432
Local anaesthetics	F, indirect, with precipitation	364
Magnesium	F	5, 31, 69, 73, 126, 129, 211, 279, 435, 444, 547, 574, 622, 629, 631, 642, 643

Table 3 (*continued*)

Species	Detection* and FI contribution	References
	with ion exchange	134
	with dialysis	296, 297
	with dilution	100, 608
	with slurry sampling	32, 74, 313, 334, 560, 561
	with on-line digestion	619
Manganese	F	4, 112, 203, 240, 290, 391, 425, 445, 627, 631
	with ion exchange	242, 248, 615
	with liquid–liquid extraction	12
	with electrolytic deposition	37
	with slurry sampling	32, 311, 561
	with calibration	249
	ET	30, 40
Methadone	F, indirect	366
Mercury	CV	84, 121, 150, 162, 224, 225, 229, 232, 268, 347, 350, 372, 397, 410, 438, 450, 463, 472, 473, 492, 493, 578, 632
	with ion exchange	134, 633
	with sorbent extraction	147, 217
	with speciation	55, 217, 371, 575
	with digestion	226, 233, 491, 517, 518, 583
	ET, with *in situ* collection	322, 472, 473
Molybdenum	F	533
	with ion exchange	259
Nitrate/nitrite	F, indirect with liquid–liquid extraction	208, 469
Nickel	F	37, 49, 370, 467
	with ion exchange	164, 166, 252, 326, 416, 615
	with sorbent extraction	23, 393
	wth precipitation	353
	with coprecipitation	141, 581
	ET	
	with sorbent extraction	409, 476
	with liquid–liquid extraction	25, 27
	with coprecipitation	189
Ondanstron	F, indirect	626
Oxalate	F, indirect, with precipitation	161
Perchlorate	F, indirect, with extraction	206
Palladium	ET, with sorbent extraction	289
Platinum	ET	
	with sorption	96, 289
Potassium	F	8, 31, 67, 69, 113, 129, 158, 204, 296, 297, 379, 439, 629, 631, 642, 643
	with liquid–liquid extraction	258
	with calibration	167, 622
	with dilution	100, 126
	with slurry sampling	32, 73, 74, 334
Rhodium	ET	30, 289
Reducing sugars	F, indirect	614
Selenium	ET	13, 52, 323
	HG	52, 88, 104, 105, 140, 212, 222, 224, 255, 292, 293, 302, 348, 350, 380, 382, 406, 451, 453, 494, 559, 565, 566, 567, 580, 611
	with ion exchange	600, 601, 634
	with pre-reduction and speciation	197, 354
	with coprecipitation	498
	with interference isolation	388, 389
	with digestion	407, 515
	using bound hydroborate	97
Silver	F	641
	with precipitation	153, 157
	with coprecipitation	115, 400, 401
	with liquid–liquid extraction	495
Sodium	F	10, 31, 32, 67, 69, 73, 74, 129, 154, 167, 296, 379, 629, 631, 643
	with dilution	100, 113, 126, 642
	with dialysis	297
	with calibration	8
	ET	29, 30
Strontium	F	637
Sulfonamides	F, indirect, with precipitation	365
Sulfate	F, indirect, with precipitation	108, 210, 645
Sulfide	F, indirect, with precipitation	404
Surfactants	F, indirect, with liquid extraction	209, 344
Thallium	F, with precipitation	363
Tellurium	HG	224, 611
	with digestion	515

Table 3 (*continued*)

Species	Detection* and FI contribution	References
Thiosulfate	F, indirect	155
Titanium	ET	87
Tin	HG	346, 460, 464, 586
	with ion exchange	188
	with digestion	515
	ET, with *in situ* collection	499, 630
Uranium	F, indirect	337
Vanadium	F	399
	ET	29, 30
Zinc	F	4, 18, 19, 21, 37, 46, 69, 71, 75, 80, 81, 83, 120, 122, 159, 213, 240, 311, 414, 419, 431, 470, 560, 561, 631
	with ion exchange	134, 159, 165, 247, 248, 326, 394, 395, 414, 419, 615
	with sorbent extraction	147, 327
	with digestion	76, 79, 81, 83
	with *in vivo* sampling	79
	with slurry atomization	122, 311, 560
	ET, with liquid–liquid extraction	25

* F = Flame; ET = electrothermal; HG = hydride generation; and CV = cold vapour.

higher degree of success when compared with conventional batch operations. In addition to the advantages quoted above, relative detection limits were improved by as much as an order of magnitude owing to significant reductions in detector noise level produced by a more stable argon–hydrogen flame.[224]

FI SAMPLE INTRODUCTION FOR ELECTROTHERMAL (ET) AAS

FI sample introduction for ETAAS systems is less straightforward than for FAAS or VGAAS owing to the discontinuity of ETAAS operations, this obviously being the cause of the slower development of the related techniques. However, the main obstacles for efficiently and harmoniously combining FI with ETAAS have been overcome, and with commercialized dedicated hardware and software becoming available, FI-ETAAS will experience a faster development in the future.

Most applications in FI-ETAAS are associated with on-line preconcentration; for this reason, the sample introduction techniques involved are dealt with under Sensitivity Enhancement. Apart from these, FI introduction based on thermospray[29,30] and high-pressure nebulization,[50] which can be additionally coupled to on-line preconcentration systems, shows considerable potential for future development.

FI DILUTION SYSTEMS

Owing to the relatively narrow dynamic response range of the AAS technique, dilution operations often constitute an integral part of AAS determinations, particularly when dealing with samples with high analyte concentrations, which vary within wide ranges. Being a technique based on reproducible sample dispersion (dilution), FI appears to be ideally suited for accomplishing such tasks automatically and efficiently. For this reason, a multitude of different approaches have been proposed for achieving various degrees of sample dilution, the main contributions of which are summarized in Table 4. While fixed dilution factors below 20-fold are readily achievable in a basic FI system by choosing the appropriate sample volume and other flow parameters, higher dilution factors reaching, *e.g.*, 1000 or more, often require more specialized equipment, particularly when high sample throughput, reproducibility and flexibility in choice of dilution ranges are desired. Although some methods come close to these requirements, the rather limited acceptance of FI dilution systems for routine applications suggests the need for further improvement and/or commercialization of dedicated equipment and/or related software support.

SENSITIVITY ENHANCEMENT

FI provides a multitude of ways for enhancing the sensitivity of AAS determinations. The various possibilities with their typical achievable enhancement factors reflecting the state-of-the-art compared with conventional operations under normal conditions are shown in Table 5. Almost all of the approaches are achievable in the batch operation mode; however, the unique advantage of the FI system is the high efficiency, both

Table 4 FI dilution systems for FAAS

Dilution system	Readout mode	Special equipment	s_r (%)	Frequency/h	Maximum dilution factor	Ref.
Single mixing coil	Maxima	None	0.5–1	100–200	30–40	
Merging-flows	Maxima	None	0.5–2	100–200	10	622
Split-flow	Maxima	Special manifold	1–3	100–150	30	360
Multi-line network	Maxima	Dual-valve network	0.7–3	Variable	30–40	535
Mixing chamber	Maxima/gradient	Chamber/stirrer	0.6–2	30–100	100	525
Microsampling	Maxima/area	Computer and stepper motor-driven pumps	0.6–2	60–100	1500	192
Zone sampling	Maxima	Automated dual-valve system	1–3	60–100	100	422
Zone penetration	Maxima/minima	Automated dual or 8-channel valve	0.6–2	100–2000	50	623
Micro-zone penetration	Maxima/minima	Computer and stepper motor-driven pumps	1–2	45–60	30 000	608

* s_r = Relative standard deviation.

Table 5 Sensitivity enhancement factors (relative) of FI-AAS techniques. Sampling frequencies: 60 h^{-1} (FAAS, VGAAS); 20 h^{-1} (ETAAS)

Technique	FAAS	VGAAS	ETAAS
Preconcentration:			
sorption	40–60	15–25	20–25
coprecipitation	50–60	24	20–25
solvent extraction	40–60	NA*	20–25
hydride generation	NA	NA	50–100
Donnan dialysis	10	NA	NA
Organic solvent effect	2–3	NA	NA
Slotted quartz tube	3–5	NA	NA

* NA = Not available.

in terms of throughput and reagent/sample economy, with which these can be accomplished. Except where extremely high enrichment factors are pursued in on-line preconcentration systems, the enhancement effects can currently be realized at sample throughputs approaching those of conventional AAS procedures.

Among the various approaches for sensitivity enhancement, on-line preconcentration systems have in the last decade undoubtedly enjoyed the highest popularity. This can be seen from the number and percentage of related publications accumulated mainly within this period (Fig. 3). On-line preconcentration is the only enhancement technique that has been implemented in all three main areas of AAS, *i.e.*, FAAS, ETAAS and VGAAS. In fact, the field has evolved into one of the most active research fields in FIA and atomic spectrometry. Some noteworthy developments in this field might include the following:

(a) The earlier on-line ion-exchange column preconcentration systems were rapidly supplemented by more selective preconcentration systems based on sorbent extraction with reversed-phase C_{18} silica-based sorbents.[179,442] More recent developments include the use of polymeric[283,603] and fibrous sorbents and activated carbon[373,405] in on-line columns and the sorption of ion-pairs[305,500] for further enhancing the performance of such systems.

(b) Some general principles in the design of on-line column preconcentration AAS systems for achieving high efficiency now appear to be well-established. These include mainly: time-based sample loading, countercurrent elution of the column, and the use of multiport valves for accommodating the micro-column in a loop.[182] More recent developments suggest the introduction of small air segments between sample and eluent or the use of air-flow transportation to limit dispersion between the different zones with the aim of improving the enrichment factors or decreasing the eluate volume which is particularly important in ETAAS applications.[423,588,604]

(c) Filterless on-line coprecipitation systems using knitted reactors and organic precipitants significantly improved the tolerance to interferences of preconcentration methods. Minimum effort is required for calibration when analysing a large variety of samples using the same set of experimental conditions. Thus, the same FI on-line coprecipitation FAAS method and simple aqueous calibration series were used for the determination of a number of heavy metals in biological samples with different matrices, including blood, serum, urine, and animal and plant tissues.[581]

(d) The recent commercialization of equipment for on-line column preconcentration (solid-phase extraction) definitely further enhances the propagation of such techniques in the AA laboratory.

An important trend in enhancing sensitivity is the synergistic combination of multiple enhancement effects in a single FI-AAS system to achieve large enhancement effects with high efficiency. FI has proved to be an ideal vehicle for achieving such goals. An example is that reported by our group[190] where on-line coprecipitation preconcentration, organic solvent effect, and slotted quartz tube atom retarded effects were combined in an FI-FAAS system, achieving a 250-fold enhancement for lead at a sample throughput of 72 h^{-1}.

SPECIATION

The on-line separation capabilities provided by FI systems have been exploited also for speciation purposes, often with preconcentration effects at least for one of the separated species. Most studies were based on column techniques where differences in retention properties were utilized,[356,478,482] but differences in reaction kinetics were also made use of, particularly in hydride generation (HG) AAS systems.[123,611] FI was also shown to be an ideal interface for coupling liquid chromatographs to the AA spectrometer in speciation studies.[427] Such studies are expected to increase in the future.

FI-FAAS INDIRECT DETERMINATIONS

This is one of the fields in which the benefits of FI-FAAS are best demonstrated. A multitude of organic analytes which cannot be determined by AAS directly, or even indirectly under batch operation conditions, have been efficiently determined by FI-AAS *via* the measurement of appropriate tag-elements. Valcárcel's group in Cordoba[548] has undoubtedly contributed most to the development of this field.

Most of the methods are based on chemical reactions involving precipitate or complex formation between the analyte and the tag-metal, and subsequent AA measurement of the latter following a suitable separation procedure (*e.g.*, on-line filtration, liquid–liquid extraction).[209,230,366,369] More recently, solid-phase reactions involving the release of tag-metals from on-line columns packed with immobilized reagents have shown promise by producing highly efficient methods for a number of pharmaceutical constituents.[218] The selectivities of indirect methods are rarely satisfactory for the determination of analytes in samples with complicated and variable matrices; however, pharmaceutical products are at least one field where such limitations are not serious.[552]

ON-LINE SAMPLE DIGESTION

The perspective of achieving on-line digestion of samples for AAS measurement as first shown by Burguera and Burguera[81] in 1986 appears particularly attractive owing to the tedious and labour-intensive nature of digestion procedures. With a few exceptions, such applications almost always involve the use of microwave ovens for achieving rapid heating either in a continuous-flow or in stopped-flow mode. With commercialized small-capacity microwave heating equipment becoming available the interest in such applications is growing rapidly. However, hitherto most of the applications were applied to liquid samples such as blood, serum and urine, often with a need for matching the sample matrix in calibration standards.[79] One of the successful applications is that reported by Welz *et al.*[583] for the determination of mercury in urine where eight different organic and inorganic mercury species were quantitatively recorded, using simple aqueous standards for calibration. Digestion of solid samples, including plant and animal tissues and coal, in the form of slurries has also been attempted.[99,220,221,239] However, the degree of success seems to depend on the nature of the samples digested. Coal samples, for example, were not completely digested even in a pressured stopped-flow digestion system.[221]

Table 6 FI-FAAS calibration systems

Single standard	Ref.	Multiple standards	Ref.
Gradient methods:		Peak width method	527
Exponential flask	521	Interpolative*	521
Zone penetration*	167	Merging flow*	331
Gradient ratio	475		
Gradient dilution	214		
Gradient dilution*	8		
On-line serial dilution:			
mixing coil series	535		
microsampling	195		

* Standard additions method.

CALIBRATION SYSTEMS

FAAS is undoubtedly a field on which the largest efforts for the development of FI calibration systems has been focused, as demonstrated by the diversity of related approaches available. The methods may be grouped into those requiring a single standard and those that still require the preparation of a set of standards. The attractiveness of the former is obvious, and more efforts have been devoted to this direction. This involves either exploitation of the reproducible concentration gradient of FI peak profiles or automated stepwise dilution of a top standard to produce a calibration series. The main FI calibration methods for FAAS are listed in Table 6. Despite the relatively large number of choices available and the ingenuity in the design of many approaches, hitherto none seemed to have reached a stage for routine use or for commercialization. At this stage, further efforts on refinement are required to produce more robust and reliable systems which are supported by dedicated computer software.

CONCLUSIONS

The foregoing decade of development of FI-AAS was flagged with multitudinous accomplishments which have brought much excitement to researchers in this field. By inducing stronger ties between chemistry and atomic spectrometry, the combined technique has stimulated a new era of development for AAS, and atomic spectrometry in general, which is expected to be carried on into the next century. What has been experienced in FI-AAS is bound to be repeated in FI-ICP-ES, FI-AFS and ICP-MS in the future.

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